State of California California Environmental Protection Agency AIR RESOURCES BOARD

APPENDICES

FOR THE

Report for the Air Monitoring of EPTC in Merced County (Application) and in Imperial County (Ambient)

Engineering and Laboratory Branch

Monitoring and Laboratory Division

Project No. C97-021 (Application) C96-035 (Ambient)

Date: June 10, 1998

APPENDIX I SAMPLING PROTOCOL



Environmental Protection

Air Resources Board

P.O. Box 2815 2020 L Street

Sacramento, CA 95812-2815

Agency

MEMORANDUM

Dr. John Sanders, Chief

Environmental Monitoring and Pest Management Branch

Department of Pesticide Regulations

James M. Strock Secretary for Environmental Protection

FROM:

TO:

George Lew, Chief / Engineering and Laboratory branch

Monitoring and Laboratory Division

DATE:

October 10, 1996

SUBJECT: FINAL EPTC MONITORING PROTOCOL

Enclosed is the final monitoring protocol, "Protocol for the Ambient Air Monitoring of EPTC in Imperial County During Fall, 1996." The draft "Standard Operating Procedures for the Analysis of EPTC in the Ambient Air" is included in the protocol as Attachment B.

If you or your staff have questions or need further information, please contact me at 263-1630 or Kevin Mongar at 263-2063.

Enclosure

cc: Genevieve Shiroma, SSD (w/attachment)

Jeff Cook, MLD (w/attachment)

Linda Evans, Imperial County Ag Comm. Office (w/attachment)

Stephen Birdsall, ICAPCD (w/attachment)

State of California California Environmental Protection Agency AIR RESOURCES BOARD

Protocol for the Ambient Air Monitoring of EPTC In Imperial County During Fall, 1996

Engineering and Laboratory Branch

Monitoring and Laboratory Division

Project No. C96-035

Date: October 7, 1996

APPROVED:

Nin Mongar, Project Engineer

Cynthia L. Castronovo, Manager

Testing Section

George Lew, Chief

Engineering and Laboratory Branch

This protocol has been reviewed by the staff of the California Air Resources Board and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Board, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Protocol for the Ambient Air Monitoring of EPTC In Imperial County During Fall, 1996

I. Introduction

At the request of the California Department of Pesticide Regulation (DPR), (October 31, 1995 Memorandum from John Sanders to George Lew) the Air Resources Board (ARB) staff will determine airborne concentrations of the pesticide EPTC (dipropylcarbamothioc acid Sethyl ester) over a five week ambient monitoring program in populated areas. This monitoring will be done to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. The monitoring is in support of the DPR toxic air contaminant program and will be conducted in Imperial County.

The draft method development results and "Standard Operating Procedures for the Analysis of EPTC in Ambient Air" are included in this protocol as Attachment B.

II. Chemical Properties of EPTC

In order to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article, 1.5), the Department of Pesticide Regulation (DPR) has previously requested that the Air Resources (ARB) document the airborne concentrations of the pesticide EPTC. This recommendation provides background and recent use information on EPTC-containing products, and identifies how they are used.

EPTC (CAS: 759-94-4) is a colorless to light yellow liquid with an amine-like odor. Technical grades are yellow. EPTC has a molecular formula of $C_9H_{19}NOS$, a formula weight of 189.32 g/mole, and a specific density of 0.960 at 25°C. It has a water solubility of 375 mg/L at 25°C, a Henry's Constant of 1.0×10^{-5} atm·m³/mol at 20-25°C, and a vapor pressure of 3.4×10^{-2} mmHg at $20^{\circ}C$. EPTC is miscible with most organic solvents.

EPTC is rapidly metabolized by soil micro-organisms to carbon dioxide, mercaptan, and amino residues. Mineralization has not been reported in sterile soils due to the lack of production of carbon dioxide. Soil half-life (t_{1/2}) ranges from 4-6 weeks when applied at recommended rates. In plants, EPTC is rapidly metabolized to carbon dioxide and other naturally occurring plant constituents. EPTC sulfoxide has been reported in some soils and in corn plants.

The acute oral LD_{50} of EPTC for male rats and mice is 1,700 and 3,200 mg/kg. The LC_{50} (48 hour) for rainbow trout is 19 mg/L, and 27 mg/L for bluegill sunfish. EPTC has entered the risk assessment process at DPR under the SB 950 (Birth Defect Prevention Act of 1984) based on its potential neurotoxicity, mutagenicity, and teratologic and chronic toxicity adverse health effects.

III. Sampling

Samples will be collected by passing a measured volume of ambient air through XAD-2 resin. The exposed XAD-2 resin tubes (SKC #226-30-06) are stored in an ice chest (dry ice) or freezer until desorbed with 3 ml of ethyl acetate. The flow rate will be accurately measured and the sampling system operated continuously with the exact operating interval noted. The resin tubes will be protected from direct sunlight and supported about 1.5 meters above the ground during the sampling period. At the end of each sampling period, the tubes will be capped and placed in culture tubes with an identification label affixed. Any EPTC present in the sampled ambient air will be captured by the XAD-2 adsorbent. Subsequent to sampling, the sample tubes will be transported on dry ice, as soon as reasonably possible, to the ARB Monitoring and Laboratory Division, Testing Section laboratory for analysis. The samples will be stored in the freezer or analyzed immediately.

A sketch of the sampling apparatus is shown in Attachment A. Calibrated rotameters will be used to set and measure sample flow rates. Samplers will be leak checked prior to and after each sampling period with the sampling cartridges installed. Any change in the flow rates will be recorded in the field log book. The field log book will also be used to record start and stop times, sample identifications and any other significant data.

Ambient Monitoring

The use patterns for EPTC suggest that monitoring should occur over a 30- to 45-day sampling period in either Imperial or San Joaquin County. Sampling may be conducted during the months of October and November in Imperial County; alternatively, sampling may be conducted in San Joaquin County during April and May. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. In Imperial County, sampling sites should be located near alfalfa and sugarbeet growing areas. In San Joaquin County, sampling sites should be located near corn growing areas. Ambient samples should not be collected from samplers immediately adjacent to fields where EPTC is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during the sampling period. Background samples should be collected in an area distant to EPTC applications.

Replicate (collocated) samples are needed for five dates at each sampling location. The date chosen for replicate samples should be distributed over the entire sampling period. They may, but need not be, the same dates at every site.

Four sampling sites plus an urban background site were selected by ARB personnel from the areas of Imperial County where alfalfa farming is predominant. Sites were selected for their proximity to the fields with considerations for both accessibility and security of the sampling equipment. The five sites were at the following locations: Meadows Union School, Holtville; Imperial County Agricultural Commissioner's Office, El Centro (background); Felipe and Ramon Primary School, Heber; ICAQMD PM-10 Ambient Monitoring Station, Harris & McConnell Roads; Imperial County Fire Department, Imperial. Addresses for the sites are listed in Table 1.

	TABLE 1. Ambient Sampling Sites			
MUS	Meadows Union School S-80 at Bowker Road Holtville, CA 92250	(619) 352-7512 Larry Kelly		
ACO	Agricultural Commissioner's Office 150 S. 9th St. El Centro, CA 92243	(619) 339-4314 Janet Evans		
IHS	Imperial High School 517 W. Barioni Blvd. Imperial, CA	(619) 355-3200 Joe Maruca		
ARB	ARB Ambient Monitoring Station 1029 Ethel Calexico, CA 92231	(818) 575-6856 Curt Schreiber		
HFD	Imperial County Fire Department 1085 Ingram Heber, CA 92249 (619) 355-1191	(619) 353-0323 Ricardo Valenzuela		

The samples will be collected by ARB personnel over a five week period from October 9 - November 15, 1996. 24-hour samples will be taken Monday through Friday (4 samples/week) at a flow rate of approximately 2 L/minute.

IV. Analysis

The method development results and "Standard Operating Procedures for the Analysis of EPTC in Ambient Air" are included in this protocol as Attachment B.

V. Quality Assurance

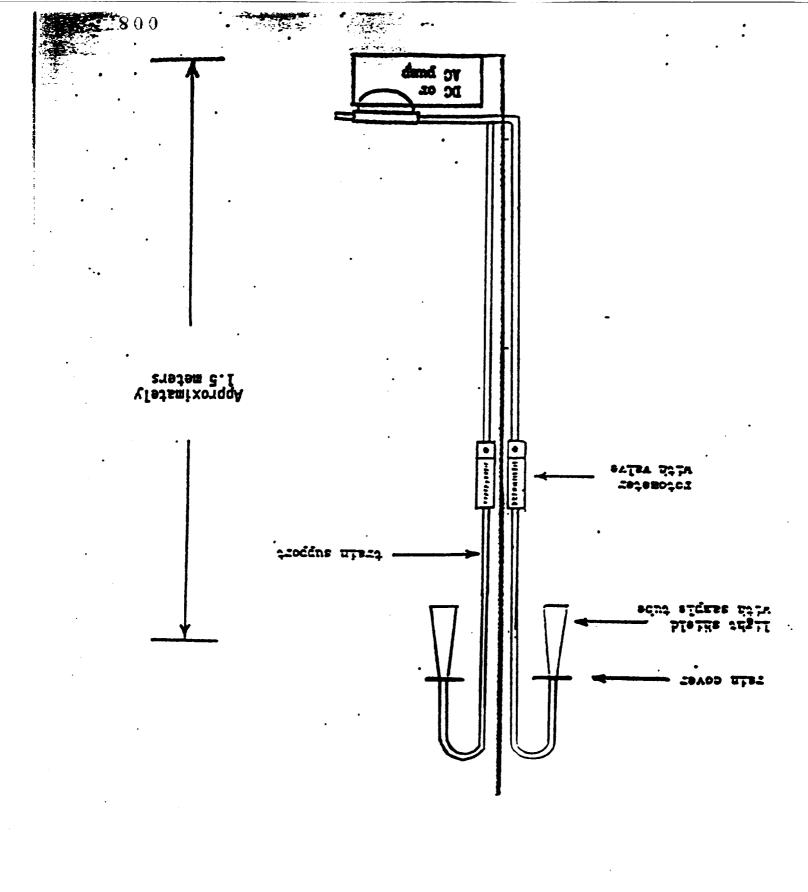
Field Quality Control for the ambient monitoring will include: 1) Five field spikes (same environmental and experimental conditions as those occurring at the time of ambient sampling) will be prepared by the Quality Management and Operations Support Branch (QMSOB) and spiked at five different levels. The field spikes will be obtained by sampling ambient air at the background monitoring site for 24 hour periods at 2 L/minute. 2) Five trip spikes will be prepared by the QMOSB and spiked at five different levels. 3) Replicate samples will be taken for five dates at each sampling location. 4) Trip blanks will be obtained at each of the five sampling locations. Procedures will follow ARB's "Quality Assurance Plan for Pesticide Monitoring" (Attachment C).

The instrument dependent parameters (reproducibility, linearity and minimum detection limit) will be checked prior to analysis. A chain of custody sheet will accompany all samples. Rotameters will be calibrated prior to and after sampling in the field.

VI. Personnel

ARB personnel will consist of Kevin Mongar (Project Engineer) and an Instrument Technician.

Attachment A



Attachment B

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State of California Air Resources Board Engineering and Laboratory Branch Monitoring and Laboratory Division

Standard Operating Procedure for the Analysis of EPTC in Ambient Air

1. SCOPE

This is a gas chromatography/mass selective detector method for the determination of EPTC from ambient air samples.

2. SUMMARY OF METHOD

The exposed XAD-2 resin tubes (\$1236-30-06) are stored in an ice chest on dry ice or in a freezer until desorbed with 3 ml of thyl acetate. A gas chromatograph (GC), using splitless injection with a DB capillary column, coupled to a mass selective detector (MSD) is used for analysis.

3. INTERFERENCES/LIMITATIONS

Method interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. A method blank must be done with each batch of samples to detect any possible method interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett Packard 5890 chromatograph

Hewlett Packard 5971 mass selective detector

Hewlett Packard 6890 Autosampler

Detector: 280°C Injector: 250°C

Column: J&W Scientific DB-35, 30 meter, 0.25 mm i.d., 0.15 um film thickness.

Program: Initial 60°C, hold 2 min, to 145°C @ 15°C/min., to 160 C @ 5 C/min., to

240 C @ 70 C/min., hold 1 min.

Column flow: He, 1.0 mL/min (8 psi @ 60 C), electronic pressure control

B. AUXILIARY APPARATUS:

- 1. Glass amber vials, 8 mL capacity.
- 2. Vial Shaker, SKC, or equiv.
- 3. Autosampler vials with septum caps.

C. REAGENTS

- 1. Ethyl Acetate, Pesticide Grade, or better
- 2. EPTC, 99% pure or better (Chem Service).

5. ANALYSIS OF SAMPLES

- 1. It is necessary to analyze a solvent blank with each batch of samples. The blank must be free of interferences. A solvent blank must be analyzed after any sample which results in possible carry-over contamination.
- 2. If a standard curve is not generated each day of analysis, at least one calibration sample must be analyzed for each parch of ten samples. The response of the standard must be within 10% of provious calibration analyses.
- 3. Carefully score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug from the primary end of the XAD-2 tube with forceps and place it into an 8 mL amber colored sample vial. Pour the XAD-2 into the vial and add 3.0 mL ethyl acetate. Retain the secondary section of the XAD-2 tube for later analysis to check the possibility of breakthrough.
- 4. Place the sample vial on a desorption shaker (or ultra sonic water-bath) for 30 minutes. Remove the EPTC extract and store in a second vial in a freezer (at -20°C) until analysis.
- 5. After calibration of the GC system, inject 2.0 ul of the extract. If the resultant peaks for EPTC have a measured area greater than that of the highest standard injected, dilute the sample and re-inject.
- 6. Calculate the concentration in ng/mL based on the data system calibration response factors. If the sample has been diluted, multiply the calculated concentration by the dilution factor.
- 7. The atmospheric concentration is calculated according to:

Conc., ng/m³ = (Extract Conc., ng/mL X 3 mL) / Air Volume Sampled, m³

6. QUALITY ASSURANCE

A. INSTRUMENT REPRODUCIBILITY

Three replicate injections of 2 uL each were made of EPTC standard solutions of order to establish the reproducibility of the instrument. This data is shown in TABLE

TABLE 1. INSTRUMENT REPRODUCIBILITY

AMOUNT INJECTED	(a)	EPTC ea count	·s)	
(ng/ml)	Average	SD	RSD	4
89	7,192	72	(±1.0%)	!
178	13,903	96	(±0.7%)	111
359	26,293	90	(±0.3%)	1
719	57,995	534	(±0.340)	10
1,437	117,368	1,424	Co Co	7

B. LINEARITY

A five point calibration curve was made using the data from TABLE 1. The corresponding linear regression equation and correlation coefficient are:

$$(y) = (82.3)(x) - (1244)$$
; Corr. Coeff. = .999

Where: y = the y-coordinate in units of area coupts

X = the x-coordinate in units of ng/ml

C. LIMIT OF DETECTION

The data above were used to calculate the limit of detection (LOD) and limit of quantitation (LOQ) for EPTC as follows:

LOD =
$$|i| + 3((i)(RSD)) = |15| + 3((15)(0.01)) = 15.5 \text{ ng/mL}$$

where: |i| = the absolute value of the x-intercept of the standard curve (ng/ml).

RSD = the relative standard deviation of the lowest concentration used for the standard curve.

$$LOQ = (3.3)(LOD) = 51 \text{ ng/ml}$$

Based on a 3 mL sample extraction volume and assuming an air sample volume of

2.7 m³ (1.9 lpm for 24 hours), the method detection limit (MDL) for ambient air sampling will be \$7 ng/cubic meter.

D. COLLECTION AND EXTRACTION EFFICIENCY (RECOVERY)

Collection and extraction efficiency data for EPTC on XAD-2 is presented in TABLE 2.

TABLE 2.
COLLECTION AND EXTRACTION
EFFICIENCY FOR EPTC ON XAD-2

EPTC			
Amount Spiked (ng)	Amount Recovered (ng)	(%)	
2156	2,070	96.0%	
2156	2,226	103%	
2156	2,190	102%	
2156	2,193	102%	
4311	3,642	84.5%	
4311	4,174	96.8%	
4311	3,836	89.0%	

The standards were spiked on the primary section of an XAD-2 tube. The tube was then subjected to an air flow of approximately 2 lpm for 24 hours. The tubes were run at an ambient temperature of approximately 85°F. The primary sections were then desorbed with 3.0 mL of ethyl acetate and analyzed by capillary column GC/MSD.

E. STORAGE STABILITY

Storage stability studies were done in triplicate for 719 ng EPTC spikes on XAD-2 tube primary sections over a period of 20 days. The percent recovery data for storage stability is presented in TABLE 3. (This section will be added later.)

TABLE 3. EPTC STORAGE STABILITY AT -20°C

PERCENT RECOVERY				
0 DAY 2 DAYS 7 DAYS 20 DAYS				

F. BREAKTHROUGH

Triplicate tubes were spiked at 4311 ng/tube then run for 24 hours at approximately 2 lpm prior to analysis. No EPTC was detected in the secondary sections of any of the three tubes.



Attachment C

State of California California Environmental Protection Agency Air Resources Board

QUALITY ASSURANCE PLAN
FOR PESTICIDE MONITORING

Prepared by the

Monitoring and Laboratory Division

and

Stationary Source Division

Revised: February 4, 1994

APPROVED:

voice Dursus, Chief

Toxic Air Contaminant Identification Branch

, Chief (Life) Management and Operations

Support Branch

MONAL FOLL . Chief Engineering Evaluation Branch

This Quality Assurance Plan has been reviewed by the staff of the California Air Resources Board and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Beard, nor does mention of trade names or commercial products constitute endersement or recommendation for use:

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QUALITY ASSURANCE PLAN FOR PESTICION MONITORING

[. Introduction

At the request of the Department of Pesticide Regulation (DPR), the Air Resources Board (ARB) documents the "level of airborne emissions" of specified pesticides. This is usually accomplished through two types of monitoring. The first consists of one month of ambient monitoring in the area of, and during the season of, peak use of the specified pesticide. The second is monitoring near a field during and after (up to 72 hours) an application has occurred. These are referred to as ambient and application monitoring, respectively. To help clarify the differences between these two monitoring programs, ambient and application are highlighted in bold in this document when the information applies specifically to either program. The purpose of this document is to specify quality assurance activities for the sampling and laboratory analysis of the monitored pesticide.

A. Quality Assurance Policy Statement

it is the policy of the ARB to provide OPR with as reliable and accurate data as possible. The goal of this document is to identify procedures that ensure the implementation of this policy.

8. Quality Assurance Objectives

Quality assurance objectives for pesticide monitoring are: (1) to establish the necessary quality control activities relating to site selection, sample collection, sampling protocol, sample analysis, data reduction and validation, and final reports; and (2) to assess data quality in terms of precision, accuracy and completeness.

II. Siting

Probe siting criteria for ambient pesticide monitoring are listed in TABLE 1. Normally four sites will be chosen. The monitoring objective for these sites is to measure population exposure near the perimeter of towns or in the area of the town where the highest concentrations are expected based on prevailing winds and proximity to applications. One of these sites is usually designated to be an urban area "background" site and is located away from any expected applications; however, because application sites are not known prior to the start of monitoring, a "zero level" background may not occur. Detectable levels of some pesticides may also be found at an urban area background site if they are marketed for residential as well as commercial use.

Probe siting criteria for placement of samplers near a pesticide application for collection of samples are the same as ambient monitoring (TABLE 1). In addition, the placement of the application samplers should be to obtain upwind and downwind concentrations of the pesticide. Since winds are variable and do not always conform to expected patterns, the goal is to surround the

notically a field with one sampler on each side (assuming the normal notice), in shape) it a distance of about 20 yards from the perimeter of the field, dowever, conditions at the site will dictate the actual placement of monitoring stations. Once monitoring has begun, the sampling stations will not be moved, even if the wind direction has changed.

[[[. Samoling

All sampling will be coordinated through the County Agricultural Commissioner's Office and the local Air Quality Management District (AQMD) or Air Pollution Control District (APCD). Monitoring sites will be arranged through the cooperation of applicators, growers or owners for application monitoring. For selection of ambient sites, ARB staff will work through authorized representatives of private companies or government agencies.

A. Background Sampling

A background sample will be taken at all sites prior to an application. It should be a minimum of one hour and longer if scheduling permits. This sample will establish if any of the pesticide being monitored is present prior to the application. It also can indicate if other environmental factors are interfering with the detection of the pesticide of concern during analysis.

While one of the sampling sites for ambient monitoring is referred to as an "urban area background," it is not a background sample in the conventional sense because the intent is not to find a non-detectable level or a "background" level prior to a particular event (or application). This site is chosen to represent a low probability of finding the pesticide and a high probability of public exposure if significant levels of the pesticide are detected at this urban background site.

B. Schedule

Samples for ambient pesticide monitoring will be collected over 24-hour periods on a schedule, in general, of 4 samples per week for 4 weeks. Field application monitoring will follow the schedule guidelines outlined in TABLE 2.

C. Blanks and Spikes

Field blanks should be included with each batch of samples submitted for analysis. This will usually require one blank for an application monitoring and one blank per week for an ambient monitoring program. Whenever possible, trip spikes should be provided for both ambient and application monitoring. The spiked samples should be stored in the same manner as the samples and returned to the laboratory for analysis.

D. Meteorological Station

Data on wind speed and direction will be collected during application monitoring by use of an on-site meteorological station. If appropriate

equipment is available, temperature and humidity data should also be collected and all meteorological data recorded on a data logger. Meteorological data are not collected for ambient monitoring.

E. Collocation

for both ambient and application monitoring, precision will be demonstrated by collecting samples from a collocated sampling site. An additional ambient sampler will be collocated with one of the samplers and will be rotated among the sampling sites so that duplicate samples are collected at at least three different sites. The samplers should be located between two and four meters apart if they are high volume samplers in order to preclude airflow interference. This consideration is not necessary for low (<20 liters/min.) flow samplers. The duplicate sampler for application monitoring should be downwind at the sampling site where the highest concentrations are expected. When feasible, duplicate application samples should be collected at every site.

F. Calibration

Field flow calibrators (rotometers, flow meters or critical orifices) shall be calibrated against a referenced standard prior to a monitoring period. This referenced standard should be verified, certified or calibrated with respect to a primary standard at least once a year with the method clearly documented. Sampling flow rates should be checked in the field and noted before and after each sampling period. Before flow rates are checked, the sampling system should be leak checked.

G. Flow Audit

A flow audit of the field air samplers should be conducted by an independent agency prior to monitoring. If results of this audit indicate actual flow rates differ from the calibrated values by more than 10%, the field calibrators should be rechecked until they meet this objective.

H. Log Sheets

Field data sheets will be used to record sampling date and location, initials of individuals conducting sampling, sample number or identification, initial and final time, initial and final flow rate, malfunctions, leak checks, weather conditions (e.g., rain) and any other pertinent data which could influence sample results.

I. Preventative Maintenance

To prevent loss of data, spare pumps and other sampling materials should be kept available in the field by the operator. A periodic check of sampling pumps, meteorological instruments, extension cords, etc., should be made by sampling personnel.

TABLE 1. PESTICIDE PROBE SITING CRITERIA SUMMARY

The following probe siting criteria apply to pesticide monitoring and are summarized from the U.S. EPA ambient monitoring criteria (40 CFR 58) which are used by the AR8.

Height Above	Minimum Distance From Supporting Structure (Meters)			
Ground (<u>Meters)</u>	<u>Vertical</u>	<u>Horizontal</u>	Other Spacing Criteria	
2-15	1	.1	1. Should be 20 meters from trees.	
٠			 Distance from sample to obstacle, such as buildings, must be a least twice the heig the obstacle protrud above the sampler. 	t
			3. Must have unrestrict air-flow 270° around	ed

sampler.

TABLE 2. GUIDELINES FOR APPLICATION SAMPLING SCHEDULE

All samplers should be sited approximately 20 yards from the edge of the field; four samplers to surround the field whenever possible. At least one site should have a collocated (duplicate) sampler.

The approximate sampling schedule for each station is listed below; however, these are only approximate guidelines since starting time and length of application will dictate variances.

- Background sample (minimum 1-hour sample: within 24 hours prior to application).
- Application + 1 hour after application combined sample.
- 2-hour sample from 1 to 3 hours after the application.
- 4-hour sample from 3 to 7 hours after the application.
- 8-hour sample from 7 to 15 hours after the application.
- 9-hour sample from 15 to 24 hours after the application.
- 1st 24-hour sample starting at the end of the 9-hour sample.
- 2nd 24-hour sample starting 24 hours after the end of the 9-hour sample.

[V. Priticil

Prior to conducting any pesticide monitoring, a protocol, using this document as a guideline, will be written by the ARB staff. The protocol describes the overall monitoring program, the purpose of the monitoring and includes the following topics:

- 1. Identification of the sample site locations, if possible.
- 2. Description of the sampling train and a schematic showing the component parts and their relationship to one another in the assembled train, including specifics of the sampling media (e.g., resin type and volume, filter composition, pore size and diameter, catalog number, etc.).
- 3. Specification of sampling periods and flow rates.
- 4. Description of the analytical method.
- 5. Tentative test schedule and expected test personnel.

Specific sampling methods and activities will also be described in the monitoring plan (protocol) for review by ARB and DPR. Criteria which apply to all sampling include: (1) chain of custody forms (APPENDIX I), accompanying all samples, (2) light and rain shields protecting samples during monitoring, and (3) storing samples in an ice chest (with dry ice if required for sample stability) or freezer, until delivery to the laboratory. The protocol should include: equipment specifications (when necessary), special sample handling and an outline of sampling procedures. The protocol should specify any procedures unique to a specific pesticide.

V. Analysis

Analysis of all field samples must be conducted by a fully competent laboratory. To ensure the capability of the laboratory, an analytical audit and systems audit should be performed by the ARB Quality Management and Operations Support Branch (QMOSB) prior to the first analysis. After a history of competence is demonstrated, an audit prior to each analysis is not necessary. However, during each analysis spiked samples should be provided to the laboratory to demonstrate accuracy.

A. Standard Operating Procedures

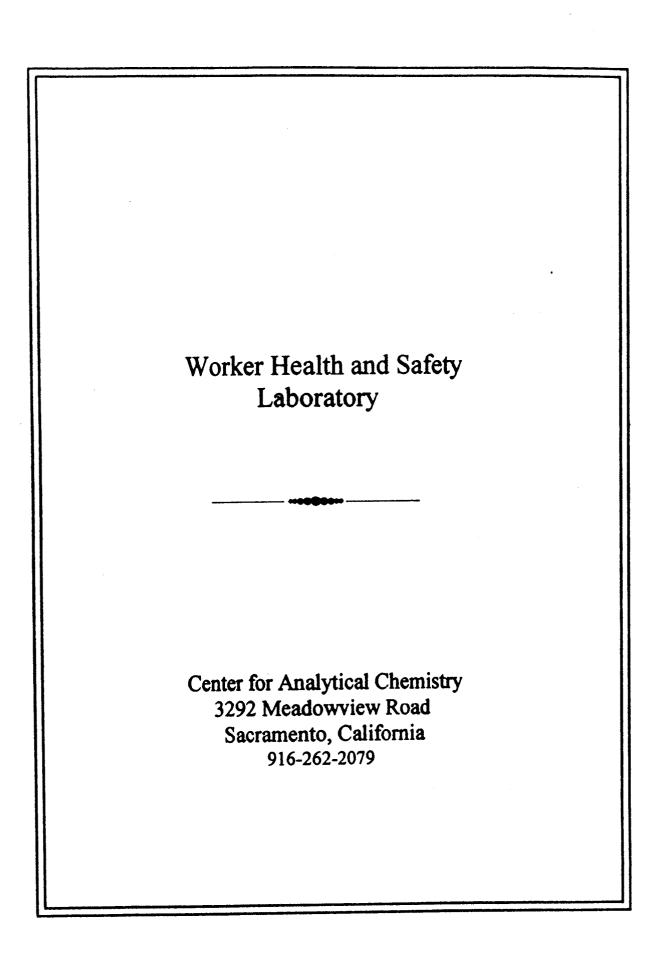
Analysis methods should be documented in a Standard Operating Procedure (S.O.P.) before monitoring begins. The S.O.P. includes: instrument and operating parameters, sample preparation, calibration procedures and quality assurance procedures. The limit of quantitation must be defined if different than the limit of detection. The method of calculating these values should also be clearly explained in the S.O.P.

much data as possible should be collected about the application conditions (e.g., formulation, application rate, acreage applied, length of application and method of application). This may be provided either through a copy of the Notice of Intent, the Pesticide Control Advisor's (PCA) recommendation or completion of the Application Site Checklist (APPENDIX II). Wind speed and direction data should be reported for the application site during the monitoring period. Any additional meteorological data collected should also be reported.

C. Quality Assurance

All quality control and quality assurance samples (blanks, spikes, etc.) analyzed by the laboratory must be reported. Results of all method development and/or validation studies (if not contained in the S.O.P.) will also be reported. The results of any quality assurance activities conducted by an agency other than the analytical laboratory should be included in the report as an appendix. This includes analytical audits, system audits and flow rate audits.

APPENDIX II LABORATORY REPORT



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	Air Sample Analysis Report	
	for	
	EPTC Application	
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	Submitted by:	
H	Sheila Margetich	
	Supervisor	
	Worker Health and Safety Laboratory	
	10-3-97	

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I. Summary of ARB/CAC Contract

The Worker Health and Safety Laboratory (WHS) of the Center for Analytical Chemistry (CAC) was contracted by the Air Resources Board to perform the analysis of air samples. In partial agreement of that contract, we analyzed one set of EPTC application samples plus accompanying QA samples.

The following Table 1 summarizes the 47 EPTC samples submitted by ARB and their analytical completion dates. Please see Attachment A1 > A3 for copies of the original chain of custody forms that accompanied these samples. The analytical results are presented in Table 2. Analyses were performed for EPTC for each sample.

TABLE 1. ARB AIR SAMPLE LOG WITH ANALYTICAL COMPLETION DATES

Date Received	ARB Logbook Numbers	Total # of air samples	Analysis
	(Inclusive)		Completion Date
5-30-97	EPTC Application 1-43	47	9-09-97

Table 2
Analytical Result Record

ARB EPTC Application	ARB Field Sample	EPTC *	WHS Lab Number
Logbook #	Number	ug/sample	
1	SB	ND	WHSC-152
3	WB	ND	WHSC-153
5	NB	ND	WHSC-154
7	EB	ND	WHSC-155
9	S1	2.92	WHSC-156
10	S1D	3.00	WHSC-157
11**	W1	ND	WHSC-160
12**	N1	ND	WHSC-162
13**	E1	ND	WHSC-164
14	S2	0.12	WHSC-158
15	S2D	0.13	WHSC-159
16**	W2	ND	WHSC-161
17**	N2	ND	WHSC-163
18**	E2	1.54	WHSC-165
19	S 3	4.05	WHSC-166
20	S3D	4.25	WHSC-167
21	W3	ND	WHSC-168
22	N3	ND	WHSC-169
23	E3	2.35	WHSC-170
24	S4	11.97	WHSC-171
25	S4D	11.38	WHSC-172
26	W4	8.74	WHSC-173
27	N4	2.55	WHSC-174
28	E4	11.87	WHSC-175
29	S 5	1.71	WHSC-176
30	S5D	1.42	WHSC-177
31	W5	1.24	WHSC-178
32	N5	2.61	WHSC-179
33	E5	7.23	WHSC-180
34	S6	1.05	WHSC-181
35	S6D	0.81	WHSC-182
36	W6	0.92	WHSC-183
37	N6	0.73	WHSC-184
38	E6_	2.49	WHSC-185
43	BLANK	ND	WHSC-186

^{*}EPTC Limit of Quantitation: 0.09 ug/sample

^{**} Labels on these samples were incorrect. Log and sample numbers were reassigned as per the ARB Project Manager.

III. Summary of WHS Analytical Report

1. SCOPE:

This report covers the WHS analysis of samples labeled EPTC Application Log #1-43 (C97-021) and associated QA samples.

2. **SUMMARY OF METHOD:**

The analytical method titled "Standard Operating Procedure for the Analysis of EPTC in Ambient Air" as supplied by the State of California Air Resources Board was followed except for 1) the GC model, 2) the detector, 3) the column, and 4) the column parameters. The samples were rotated instead of shaken. The 128 m/z ion was used for quantitation. Please see Attachment B for the method SOP.

WHS Instrumentation

Varian 3400 gas chromatograph, 8100 Autosampler

Detector: Saturn IV Ion Trap Detector (ITD)

Column: J & W Scientific DB-17, 15 meter, 0.25 mm i.d., 0.5 μ m film thickness Program: Initial 60 C, hold 1 min., to 150 C at 20 C/min., to 240 C at 40 C/min,

hold 2 min.

EPTC retention time: 5 minutes.

Column flow: He 10 psi

3. ANALYTICAL CALCULATIONS:

- A. The LOQ was the quantity of EPTC that gave a 10:1 S/N ratio. This corresponded to 0.06 ng. Using a 2 uL injection volume, and 3 mL sample, this calculates to 0.09 ug/sample. Because of the somewhat 'noisy' nature of mass chromatograms, peaks with S/N less than 10:1 are not reliably integrated.
- B. Analytical verification of LOQ: Please see Attachment C for chromatogram of a standard at the LOQ concentration.
- C. The ITD data handling system, with a group of Procedure Language programs to format the chromatograms and results, was used to compile the data. The multi-level quadratic function calibration algorithm was used to generate the calibration curve. According to the Saturn operating manual, the external standard calculation is as follows:

Amount Y = (AREA)y / (RESPONSE)y * (MULTIPLIER / DIVISOR)

where

(AREA)y is the area of sample peak y

(RESPONSE)y is the response factor (area units/ng) of y

MULTIPLIER is a constant DIVISOR is a constant

In our system, the multiplier for standards is always 1, and for samples the total volume of extract. The divisor for standards is always 1, and for samples is the uL injected. This calculation yields micrograms/sample.

Example:

Given the response factor is 40000 peak area units/ng for EPTC, the area of the unknown peak is 60000, the total volume of extract is 3 mL, and the injection volume 2 uL,

EPTC concentration = 60000 / 40000 * 3/2 = 2.25 ug/sample

4. **QUALITY ASSURANCE**:

A. Instrument Linearity and Reproducibility: Replicate injections of 2 uL were made of standards containing EPTC in order to establish the reproducibility of the Varian 3400/ITD GC/ITD system. TABLE 3 lists the peak areas of these standards and the % variation of the multiple injections.

TABLE 3. INSTRUMENT LINEARITY AND REPRODUCIBILITY

ng injected	Peak Areas	Averages
0.06 ng	3701-3933	Avg. 3787 +/- 4%
0.2 ng	11830-12725	Avg. 12253 +/- 4%
2 ng	90900-105683	Avg. 96099 - 5%, + 10%
6 ng	260817-278073	Avg. 269824 +/- 3%
10 ng	434539-472623	Avg. 459139 + 3%, -5%

B. Standard Curve Linearity and r-value: A five point calibration curve was made ranging from 0.06 ng to 10 ng EPTC. Please see Attachment D for a graph of the plotted data points. Please see Attachment E1 > E5 for chromatograms of the standards comprising the standard curve.

The following table lists the r-values for the standard curves generated during the course of analyzing the EPTC samples.

TABLE 4. STANDARD CURVE "r" VALUES DURING COURSE OF THE PROJECT

Correlation Coefficients

Date	EPTC."r" value
9-05-97	1.000
9-05-97	1.000
9-05-97	0.998
9-08-97	0.997
9-08-97	0.998
9-08-97	1.000
9-08-97	0.999
9-08-97	1.000
9-09-97	0.999
9-09-97	0.997
9-09-97	0.999
9-09-97	1.000

C. Analytical result acceptance criteria: Analytical acceptance criteria based on the linearity and reproducibility of standard curves are detailed in Attachment F, our SOP numbered WHS-AD-11 and titled "Data Generation and Reporting".

4. OUALITY ASSURANCE: (cont.)

D. Quality Assurance Spikes: ARB personnel prepared the Quality Assurance spikes for this study. The spiking level was unknown to the project chemist.

TABLE 5. QA SPIKES--µg/sample EPTC

ARB Log #	ARB ID	µg/sample EPTC
2	SFS1	1.58
4	WFS2	1.60
6	NFS3	1.55
8	EFS4	1.52
39	TS1	1.73
40	TS2	1.68
41	TS3	1.72
	TS4	1.74
	CDFA-S1	1.67
	CDFA-S2	1.7
	CDFA-S3	1.77
	CDFA-S4	1.68

Please see Attachment G1> G3 for resin Lab, Trip and Field spike chromatograms.

5. **OUALITY CONTROL:**

- A. Collection efficiencies and storage stability: For collection efficiencies and storage stability data, please refer to the method SOP as supplied by ARB (Attachment B).
- B. Resin sample/extract integrity: Once received in the lab, all of the resin samples and spikes were stored in Freezer # 27873. The temperature of this freezer is recorded manually every work day. The average temperature of this freezer during the storage of samples and spikes was -16 ° C. At no time did the temperature vary more than +/-3 ° C. In all cases, the resin samples and spikes were analyzed on the same day that they were extracted.

5. QUALITY CONTROL: (cont.)

C. On-going Quality Control spikes: The following table lists the WHS Laboratory on-going QC spike recoveries The resin tubes were spiked with 200, 1000, and 4000 ng EPTC. Please see Attachment H for a resin spike chromatogram.

TABLE 6. WHS LABORATORY ON-GOING QC Spikes--% Recovery

Date	Spike ID	Amount Spiked	% Recovery
9-02-97	902-LSP	200 ng	90
9-02-97	902-MSP	1000 ng	98
9-02-97	902-HSP	4000 ng	92
9-08-97	908-MSPA	1000 ng	87
9-08-97	908-MSPB	1000 ng	90
9-08-97	908 HSPA	4000 ng	94.5
9-08-97	908 HSPB	4000 ng	91.5
9-09-97	909 LSPA	200 ng	95
9-09-97	909 LSPB	200 ng	100
9-09-97	909-MSPA	1000 ng	89
9-09-97	909-MSPB	1000 ng	91

D. On-going Quality Control blanks: The following table lists the results of the resin blanks that were analyzed as part of the WHS Laboratory on-going QC for this EPTC study. Please see Attachment I for a chromatogram for a resin blank sample.

TABLE 7. WHS LABORATORY ON-GOING QC RESIN BLANK RESULTS

Date	Spike ID	Sample ID	EPTC
9-02-97	902 BLANK	BLANK	ND
9-08-97	908 BLANK	BLANK	ND
9-09-97	909 BLANK	BLANK	ND

6. **DISCUSSION**:

The high concentration of XAD resin co-extractives caused the system to steadily lose sensitivity, when compared to the pre-study injection reproducibility table. However, the signal-to-noise ratio did not decrease. This problem was controlled to the degree required by the data acceptance SOP by limiting the number of samples injected between standard sets to 8 or 9. The oven temperature ramp to 240 C, while not required to chromatograph EPTC or elute late peaks from the GC, also helped control this problem. Leaving the system at 220 C when not analyzing samples helped restore sensitivity between batches. This problem was not evaluated using the somewhat longer, slightly hotter temperature program in the method.

No break-through was observed in any of the study samples.

6. DISCUSSION: (cont.)

EPTC would appear to be a prime candidate for internal standard analysis. There are a family of these materials that could act as standards for each other. Because of the nearly linear standard curve, internal standard calibration would probably yield high quality data in spite of the co-extractive problem. It is also possible that a different solvent (hexane or methanol, perhaps) would minimize the co-extractives while giving acceptable recoveries.

(8)

Please see Attachment J for a chromatogram of an ARB EPTC resin sample.

0 3 5

CALIFORNIA AIR RESOURCES BOARD MONITORING & LABORATORY DIVISION P.O. Box 2815, Sacramento CA 95812

EPTC APPLICATION CHAIN OF CUSTODY

SAMPLE RECORD

Job #: C97-021

Date: 5 BOI 97
Sample/Run #:
Job name: EPTC Application

Log numbers: ____

Transfer

Transfer

Transfer

Transfer

Transfer

Transfer

ACTION Sample C	N		INITIALS	EM	METHOD OF STORAGE
	DAŢE	TIME	GIVEN BY	TAKEN BY	freezer ice or dry ice
	5/30/97	1110	KEM	3. Magatut	
				0	

LOG#	ID#	DESCRIPTION		
1	5B.			
2	5F5/.	(field spike #1)		
3	WB.		SWILL	-31¥0
4	WFSZ.	(field upike #2)		TAA
5	NB.	//	ORIGINAL DOCTUMENT	
6	NF53.	(full spike #3)	That to by Just	
7	ER.			
8	E\$54.	(Lich spike #4)	TRUE COPY OF THE	
9	٠ / 2		ORIGINAL DOCUMENT	
10	5/1.		DATE 5-30-97 INITIALS S	<u> </u>

RETURN THIS FORM TO: Kevin Mongar (916) 263-2063

CALIFORNIA AIR RESOURCES BOARD MONITORING & LABORATORY DIVISION P.O. Box 2815, Sacramento CA 95812

EPTC APPLICATION CHAIN OF CUSTODY

SAMPLE RECORD

Job #: <u>C97-021</u>

ACTION Sample Collected			INITIALS KEM		METHOD OF STORAGE
	DATE	TIME	GIVEN BY	TAKEN BY	freezer, ice or dry ice
Transfer	5/3/97	1110	KEM	S. Miangetal	
Transfer					

	LOG#	ID#	DESCRIPTION		
-	-//	W			
-	-12	W			
	13	ET	TRIT THE		
	15	57/	O: THE NENT		
ŧ	16	W2.	DATE :ALS		
1	17	W2.	TRUE COPY OF THE		
	18	67.	ORIGINAL DOCUMENT		
	20	530.	DATE _6-30-97_INITIALS_SW		

RETURN THIS FORM TO: Kevin Mongar (916) 263-2063

CALIFORNIA AIR RESOURCES BOARD MONITORING & LABORATORY DIVISION P.O. Box 2815, Sacramento CA 95812

EPTC APPLICATION CHAIN OF CUSTODY

SAMPLE RECORD

Job #: <u>C97-021</u>

Date: 5 13019 5

Sample/Run #:

Job name: FATC AMD

Log numbers: 2/-36

ACTION Sample Collected			INITIALS		METHOD OF STORAGE
·	DATE	TIME	GIVEN BY	TAKEN BY	freezer, ice or dry ice
Transfer	5/30/20	1110	KEM	3. Magta	
Transfer					

LOG#	ID#	DESCRIPTION
2/	W3.	
2 <i>2</i>	N3.	·
23	£3.	
24	54.	TRUE COPY OF THE
.5	540.	ORIGINAL DOCUMENT
26	W4.	DATE 5-30-97 INITIALS
27	N4·	
28	14.	•
.).	ري (۲)	
D	550.	

RETURN THIS FORM TO: Kevin Mongar (916) 263-2063

placed in fregu # 27837 Sm 5-30-97

CALIFORNIA AIR RESOURCES BOARD MONITORING & LABORATORY DIVISION P.O. Box 2815, Sacramento CA 95812

EPTC APPLICATION CHAIN OF CUSTODY

SAMPLE RECORD

Job #: <u>C97-021</u>

Sample/Run #:	Date: <u>5 / 국</u>	<u> </u>
Job name: Log numbers:	31-4	0/11.

ACTION Sample Collected			INITIALS KEM		METHOD OF STORAGE
	DATE	TIME	GIVEN BY	TAKEN BY	freezer, ice or dry ice
Transfer	5/30/97	1110	KEM	S. Mings	
Transfer				,	
Transfer					
Transfer		<u> </u>		·	
Transfer					
Transfer					

LOG#	ID#		DESCRIPTION
31	W5.		
32	N5.		
<i>3</i> 3	€5.		
34	3/2 ·	56.	TRUE COPY OF THE
35	356.	560.	ORIGINAL DOCUMENT
30	W5.	wb·	DATE 5-30-97 INITIALS SM
37	25.	N6.	
36	£.	E6.	
71	751.	(Trip spike #1	
- 0	757.	(Trip spike #2	

RETURN THIS FORM TO: Kevin Mongar (916) 263-2063

placed in freeze # 27837 80 5-20.00

CALIFORNIA AIR RESOURCES BOARD MONITORING & LABORATORY DIVISION P.O. Box 2815, Sacramento CA 95812

EPTC APPLICATION CHAIN OF CUSTODY

SAMPLE RECORD

Job #: <u>C97-021</u>

Sample/Run #:	Date: <u>FIE</u>	137
Job name: _ Log numbers:	EFIC	Aff.
Log	111-12	3

ACTION Sample Collected			INITIALS KE M		METHOD OF STORAGE
	DATE	TIME	GIVEN BY	TAKEN BY	freezer, ice
Transfer	Staler	2	1=41	S. Mingela	
Transfer					
Transfer					·
Transfer					
Transfer					
Transfer					

LOG#	ID#	DESCRIPTION	
4/	T53.	(Trip spike #3)	
42	TS4.	(Tup spike #3) (Tup spike #4)	
43	8.	TRUE COPY OF THE ORIGINAL DOCUMENT	
16-H	ω2-A ω2-B		
17-H	N2-H		
17-B	N2-B	Su 6-13-97 per Kenis Mangar	
18-H	EZ-A		
18-B	£2-B		

RETURN THIS FORM TO: Kevin Mongar (916) 263-2063

perced in freeze # 27837. Sm 5-30 97

State of California
Air Resources Board
Engineering and Laboratory Branch
Monitoring and Laboratory Division

Standard Operating Procedure for the Analysis of EPTC in Ambient Air

1. SCOPE

This is a gas chromatography/mass selective detector method for the determination of EPTC from ambient air samples.

2. SUMMARY OF METHOD

The exposed XAD-2 resin tubes (CKS 228-30-06) are stored in an ice chest on dry ice or in a freezer until desorbed with 3 ml of thyl acetate. A gas chromatograph (GC), using splitless injection with a DB-30 column, coupled to a mass selective detector (MSD) is used for analysis.

3. INTERFERENCES/LIMITATION

Method interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. A method blank must be done with each batch of samples to detect any possible method interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett Packard 5890 chromatograph

Hewlett Packard 5971 mass selective detector

Hewlett Packard 6890 Autosampler

Detector: 280°C

Column: J&W Scientific DB-35, 30 meter, 0.25 mm i.d., 0.15 um film thickness.

Program: Initial 60°C, hold 2 min, to 145°C @ 15°C/min., to 160 C @ 5 C/min., to

240 C • 70 C/min., hold 1 min.

Column flow: He, 1.0 mL/min (8 psi @ 60 C), electronic pressure control

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COFA	Phone 4
14º 262 1572	Fez #

8. AUXILIARY APPARATUS:

- 1. Glass amber vials, 8 mL capacity.
- 2. Vial Shaker, SKC, or equiv.
- Autosempler vials with septum caps.

C. REAGENTS

- 1. Ethyl Acetate, Pesticide Grade, or better
- 2. EPTC, 99% pure or better (Chem Service).

5. ANALYSIS OF SAMPLES

- 1. It is necessary to analyze a solvent blank with each batch of samples. The blank must be free of interferences. A solvent distribution be analyzed after any sample which results in possible carry-over contamination.
- 2. If a standard curve is not generated and day of analysis, at least one calibration sample must be analyzed for each patch of ten samples. The response of the standard must be within 10% of patched calibration analyses.
- 3. Carefully score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug from the primary and of the XAD-2 tube with forceps and place it into an 8 mL amber colored sample vial. Pour the XAD-2 into the vial and add 3.0 mL ethyl acetate. Retain the secondary section of the XAD-2 tube for later analysis to check the possibility of breakthrough.
- 4. Place the sample vial on a desorption shaker (or ultra sonic water-bath) for 30 minutes. Remove the EPTC extract and store in a second vial in a freezer (at -20°C) until analysis.
- 5. After calibration of the GC system, inject 2.0 ul of the extract. If the resultant peaks for EPTC have a measured area greater than that of the highest standard injected, dilute the sample and re-inject.
- 8. Calculate the concentration in ng/mL based on the data system calibration response factors. If the sample has been diluted, multiply the calculated concentration by the dilution factor.
- 7. The atmospheric concentration is calculated according to:

Conc., ng/m³ = (Extract Conc., ng/mL X 3 mL) / Air Volume Sampled, m³

6. QUALITY ASSURANCE

A. INSTRUMENT REPRODUCIBILITY

Three replicate injections of 2 uL each were made of EPTC standard solutions of order to establish the reproducibility of the instrument. This data is shown in TABLE

¥ 916 265 2067

TABLE 1. INSTRUMENT REPRODUCIBILITY

AMOUNT INJECTED	EPTC (area counts)			
(ng/ml)	Average	SD	RSD	2
89	7,192	72	(±1.0%)4	(
178	13,903	96	L±0.7%	10
359	26,293	90	下0.3	/
719	57,995	534	LOD	10
1,437	117,368	1,424	Section 1	
				-

8. LINEARITY

A five point calibration curve was made using the detairon TABLE 1. The corresponding linear regression equation and correlation coefficient are:

$$(y) = (82.3)(x) - (1244);$$
 Corr. Coeff. = .992

Where: y = the y-coordinate in units of area odpets

X = the x-coordinate in units of ng/ml

C. LIMIT OF DETECTION

The data above were used to calculate the limit of detection (LOD) and limit of quantitation (LOQ) for EPTC as follows:

$$LOD = |i| + 3((i)(RSD)) = |15| + 3((15)(0.01)) = 15.5 \text{ ng/mL}$$

where: |i| = the absolute value of the x-intercept of the standard curve (ng/ml).

RSD = the relative standard deviation of the lowest concentration used for the standard curve.

$$LOQ = (3.3)(LOD) = 51 \text{ ng/ml}$$

Based on a 3 mL sample extraction volume and assuming an air sample volume of

2.7 m³ (1.9 lpm for 24 hours), the method detection limit (MDL) for ambient air sampling will be p3-ng/cubic meter.

D. COLLECTION AND EXTRACTION EFFICIENCY (RECOVERY)

Collection and Expection efficiency data for EPTC on XAD-2 is presented in TABLE 2.

TABLE 2.
COLLECTION AND EXTRACTION
EFFICIENCY FOR EPTC ON XAD-2

EPTC			
Amount Spiked (ng)	Amount Recovered (ng)	(%)	
2156	2,070	96.0%	
2156	2,226	103%	
2156	2,190	102%	
2156	2,193	102%	
4311	3,642	84.5%	
4311	4,174	96.8%	
4311	3,836	89.0%	

The standards were spiked on the primary section of an XAD-2 tube. The tube was then subjected to an air flow of approximately 2 lpm for 24 hours. The tubes were run at an ambient temperature of approximately 85°F. The primary sections were then described with 3.0 mL of ethyl acetate and analyzed by capillary column GC/MSD.

E. STORAGE STABILITY

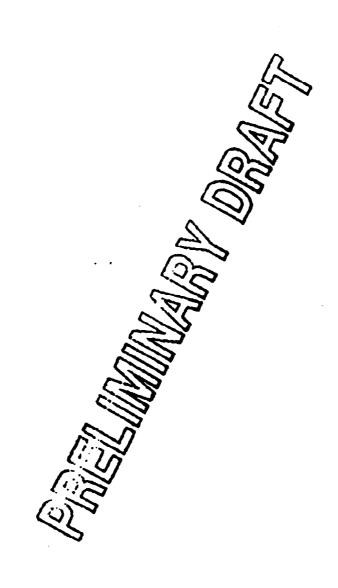
Storage stability studies were done in triplicate for 719 ng EPTC spikes on XAD-2 tube primary sections over a period of 20 days. The percent recovery data for storage stability is presented in TABLE 3. (This section will be added later.)

TABLE 3. EPTC STORAGE STABILITY AT -20°C

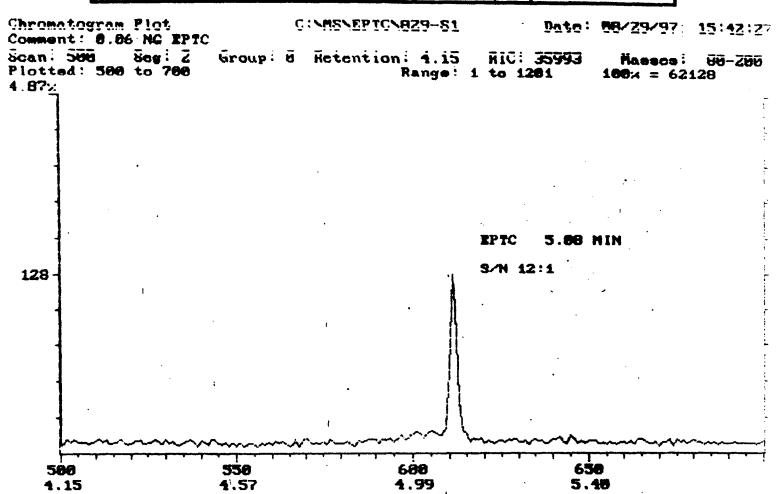
PERCENT RECOVERY			
0 DAY	2 DAYS	7 DAYS	20 DAYS

F. BREAKTHROUGH

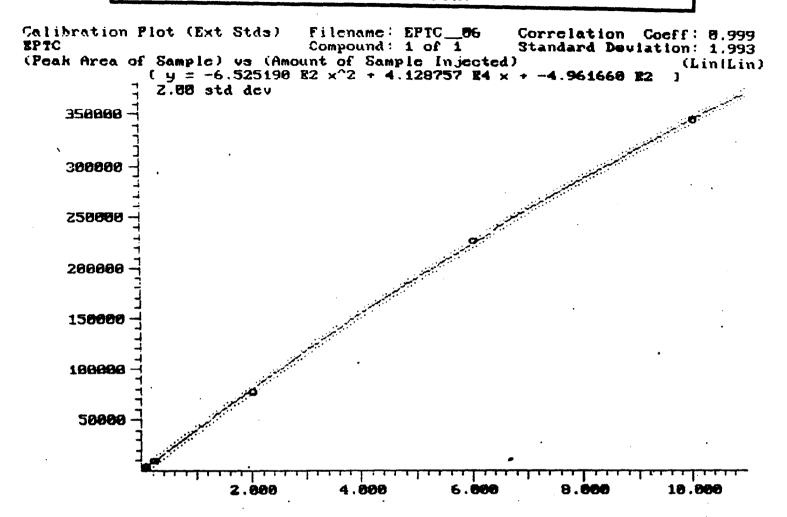
Triplicate tubes were spiked at 4311 ng/tube then run for 24 hours at approximately 2 lpm prior to analysis. No EPTC was detected in the secondary sections of any of the three tubes.



ATTACHMENT C LOQ Verification Chromatogram - 0.09 ug/sample



ATTACHMENT D Standard Curve Plot



ATTACHMENT E-1 Standard Curve Chromatogram - 0.06 ng

Comment

: 0.06 NG EPTC

Data file MLs sample UL injected

: 908-S16 : 1.000 : 1.000

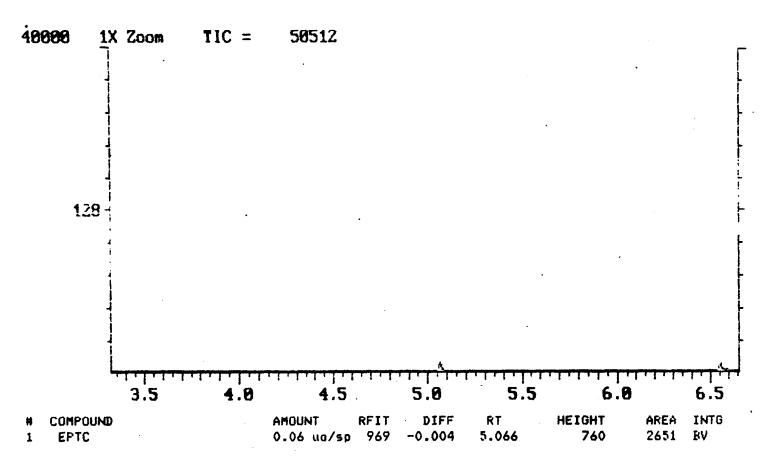
Acquired: 09 Sep 1997 7:39 am

Calibration file : EPTC__XX

Quantitation file: 908-S16

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 09 Sep Plot Spooler screen Injections... Septum 15M DB-17 6169524B 10PSI SCOTT

II S/N 036415420 1997 7:51 am 245 199 , Insert/pre-column 199 , Column 1837



ATTACHMENT E-2 Standard Curve Chromatogram - 0.2 ng

Comment

: 0.2 NG EPTC USIS 128

Data file mLs sample uL injected

908-S17 1.000 1.000

Acquired: 09 Sep 1997 7:58 am

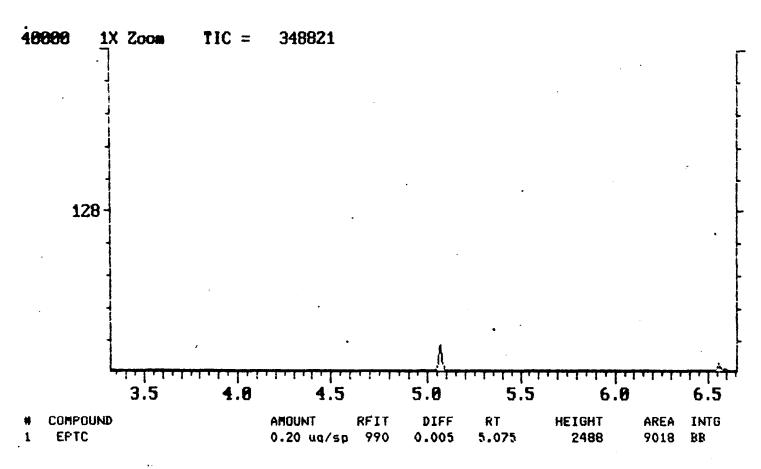
Calibration file : EPTC__XX

Quantitation file: 908-S17

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 09 Sep Plot Spooler screen Injections... Septum

15M DB-17 6169524B 10PSI SCOTT Saturn II S/N 036415420 09 Sep 1997 8:10 am II S/N 036415420 1997 8:10 am 248 200 , Insert/pro

, Insert/pre-column 200 , Column 1838



ATTACHMENT E-3 Standard Curve Chromatogram - 2.0 ng

Comment

: 2.0 NG EPTC USIS 128 ION

Data file mLs sample uL injected

: 908-S18

Acquired: 09 Sep 1997 8:17 am

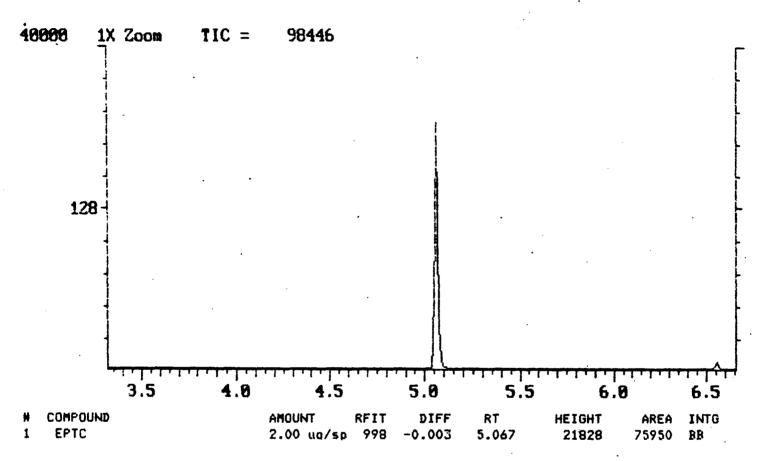
Calibration file : EPTC__XX

Quantitation file: 908-S18

Column : 15M DB Chemist : SCOTT Instrument : Saturn Date printed : 69 Sep Plot Spooler screen Injections... Septum

15M DB-17 6169524B 10PSI SCOTT

II S/N 036415420 1997 8:29 am 251 201 , Insert/pre-column 201 , Column 1839



ATTACHMENT E-4 Standard Curve Chromatogram - 6.0 ng

Comment

: 6 NG EPTC USIS 128 ION

Data file mLs sample uL injected

908-S19 1.000 1.000

Acquired: 69 Sep 1997

8:36 am

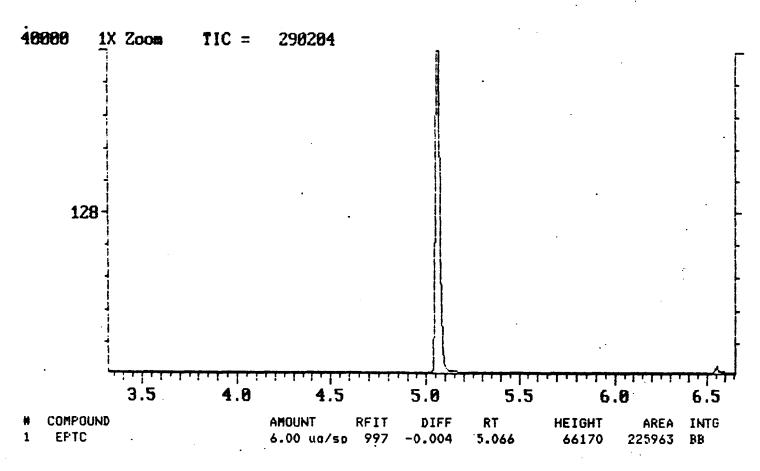
Calibration file : EPTC__XX

Quantitation file: 988-S19

15M DB-17 6169524B 10PSI SCOTT Saturn II S/N 036415420 09 Sep 1997 8:47 am creen 254 Septum 202 , Insert/pre-column

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 09 Sep Plot Spooler screen Injections... Septum

202 , Column 1840



ATTACHMENT E-5 Standard Curve Chromatogram - 10.0 ng

Comment

: 10 NG EPTC USIS 128 ION

Data file mLs sample uL injected

: 908-S20 : 1.000 : 1.000

Acquired: 09 Sep 1997 8:55 am

Calibration file : EPTC_XX

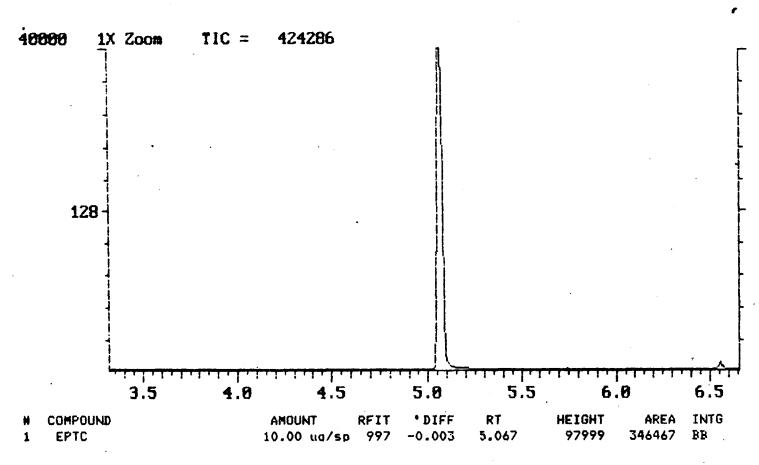
Quantitation file: 908-S20

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 09 Sep Plot Spooler screen Injections... Septum

15M DB-17 6169524B 10PSI SCOTT

II S/N 036415420 1997 9:06 am 257

203 , Insert/pre-column 203 , Column 1841



ATTACHMENT F WHS SOP - AD - 11

California Department of Food and Agriculture Center for Analytical Chemistry Worker Health and Safety Laboratory 3292 Meadowview Road Sacramento, CA 95832 Number: WHS-AD-11 Date: 02/05/96 Revision: Replaces: Page: 1 of 3

STANDARD OPERATING PROCEDURE

Title: Data Generation and Reporting

Purpose: To Provide a Standardized Procedure for the Generation and Reporting of Chromatographic Data

Scope: All laboratory personnel.

Procedure:

Any conflict with instructions in the method or protocol must be resolved with senior staff, the study director, and documented before proceeding.

The number of standards used should adequately describe the standard curve shape. Typically this is 3-5 points spanning 1-2 orders of magnitude for linear systems. For non-linear systems, additional points or narrower concentration ranges may be needed. Calibration curves should include a data point near the instrument MDL of the compound(s), or a point that approximates the project LOD. All samples with responses higher than the upper limit of the standard curve must be diluted and reanalyzed.

The number and concentration of standards necessary to "adequately describe" the curve shape depend on the type of curve fitting used for data analysis as well as the actual shape of the curve, which in turn depends on the detector used and the chemical being analyzed. In the case of point-to-point curve fitting (used by HP 5880 and 3396 integrators), the number of standards and their concentrations should be chosen so that the maximum quantitative error between a smooth curve and the point-to-point line, measured at the midpoint between consecutive standard levels, is 15% or less. Curve-fit errors in systems that can use quadratic functions (HP MSD, Varian Saturn) are much less, and consequently wider concentration ranges can be used.

In general, using peak heights for GC data will minimize errors because it reduces the effect of small leading or trailing peak interferences. For LC work, peak areas yield better data because of the tendency for LC peaks to widen and shorten during a run due to the effect of developing column voids.

Retention times should be reproducible to better than 1% in most cases for both LC and GC. Capillary GC and gradient LC times should be even better. Some systems will

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slowly drift due to changing ambient conditions in the lab, but consecutive runs should show very small changes.

Samples must be run in groups small enough that the standard curves on either side of them will not vary by more than +/- 15%. Sufficient data should be generated during method development to provide guidance for the chemist on this number, and that information should be included in the method. Typically, no more than 10-20 samples should be analyzed between standard curves. 'Conditioning' samples and cooling GC analytical systems between batches may provide more consistent data.

Residues are generally reported in micrograms/sample. In the absence of complicating factors, levels should be reported as follows:

>= 1000 ugs	to nearest 10 ug
100 to 999 ugs	to nearest ug
10 to 99.9 ugs	to nearest 0.1 ug
1 to 9.99 ugs	to nearest 0.01 ug
0.010 to .999 ugs	to nearest 0.001 ug

To prevent confusion when reporting high levels of residue, do not mix reporting units. That is, do not report some values as ugs/sample, and some as mgs/sample within the same group of samples, unless the unit changes are *clearly* marked to draw the reader's attention.

Recovery data should be reported, but sample results NOT corrected for recovery. If corrected results are reported, a notation explicitly stating that fact should be included on the report sheet.

Written By:

Scott Fredrickson, Ag. Chemist III / Worker Health & Safety Laboratory

WHS-AD-11 Revision: Page: 3 of 3

Reviewed By:

Terry Jackson, QA Officer Center for Analytical Chemistry

Approved By:

Lilia Rivera, Program Supervisor Center for Analytical Chemistry

Approved By:

William Cusick, Chief

as Yuly 91

Center for Analytical Chemistry

ATTACHMENT G-1 Resin Lab Spike Chromatogram

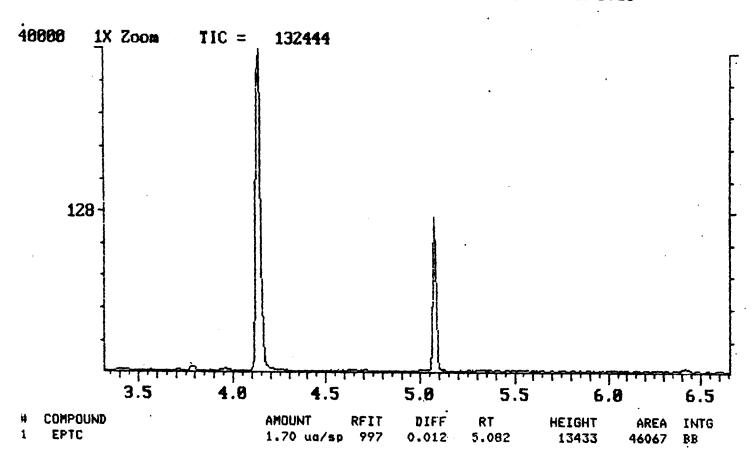
Comment : UHS 34 EPTC ANALYSIS

Data file mLs sample uL injected Acquired: 03 Sep 1997 8:53 pm

Calibration file : EPTC__XX Quantitation file: 903-34

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 03 Sep Plot Spooler screen Injections... Septum 15M DB-17 6169524B 10PSI SCOIT Saturn II S/N 036415420 03 Sep 1997 9:05 pm

67 , Insert/pre-column 67 , Column 1705



ATTACHMENT G-2 Resin Trip Spike Chromatogram

Comment

: UHS 32 EPTC ANALYSIS

Acquired: 03 Sep 1997 8:26 pm

Data file MLs sample uL injected

Calibration file : EPTC_XX

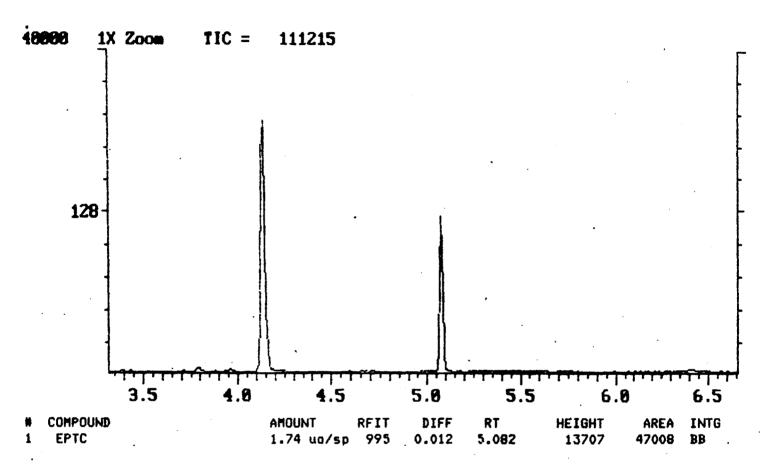
Quantitation file: 983-32

5M DB-17 6169524B 10PSI

Column : 15M DB-Chemist : \$COTT Instrument : \$aturn Date printed : 63 Sep Plot Spooler screen Injections... Septum

II S/N 036415420 1997 8:37 pm 187 65 , Insert/pre-column

65 , Column 1763



ATTACHMENT G-3 Resin Field Spike Chromatogram

Comment : UHS 25 EPTC ANALYSIS

Data file mLs sample uL injected 903-25 3.000 2.000

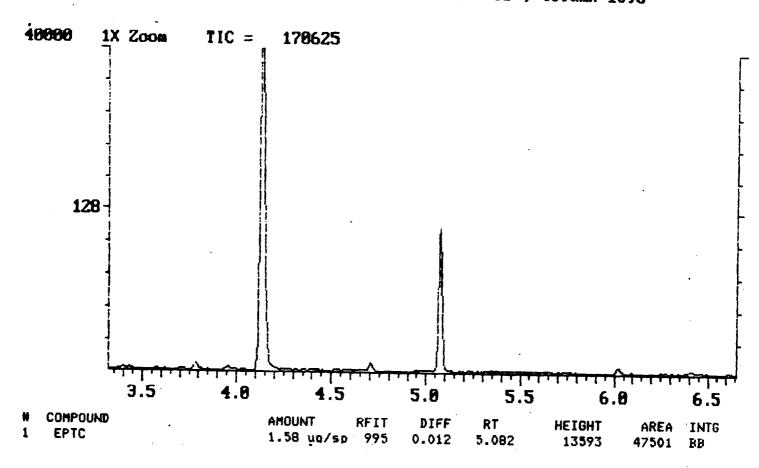
Acquired: 03 Sep 1997 5:28 pm

Calibration file : EPTC_XX

Quantitation file: 903-25

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 03 Sep Plot Spooler screen Injections... Septum 15M DB-17 6169524B 10PSI SCOTT Saturn II S/N 036415420 03 Sep 1997 5:39 pm II S/N 036415420 1997 5:39 pm 148 52 , Insert/pre-column

52 , Column 1698



ATTACHMENT H Resin On-going QC Spike Chromatogram

: 9-09 1.0 UG SPK B EPTC ANALYSIS Comment

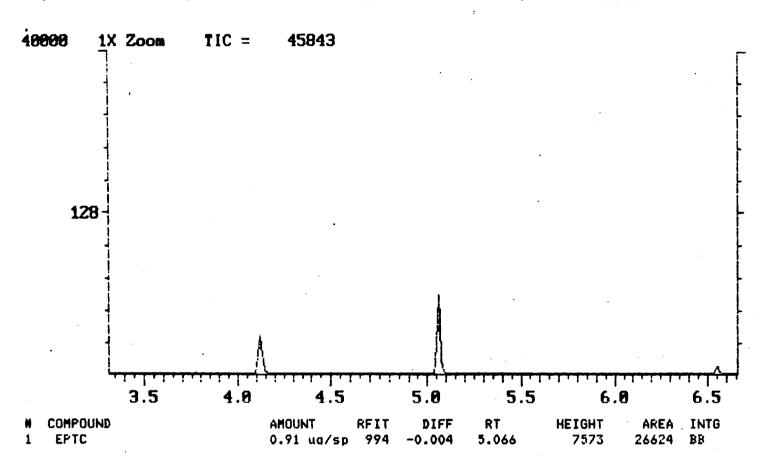
Acquired: 89 Sep 1997 5:33 pm

Data file mLs sample uL injected 909-MSPB 3.000 2.000

Calibration file : EPTC_XX Quantitation file: 909-MSPB

Column : 15M DB Chemist : SCOTT Instrument : Saturn Date printed : 09 Sep Plot Spooler screen Injections... Septum 15M DB-17 6169524B 10PSI SCOTT Saturn II S/N 036415420 09 Sep 1997 5:44 pm

, Insert/pre-column 227 , Column 1865



ATTACHMENT [Resin On-going QC Blank Chromatogram

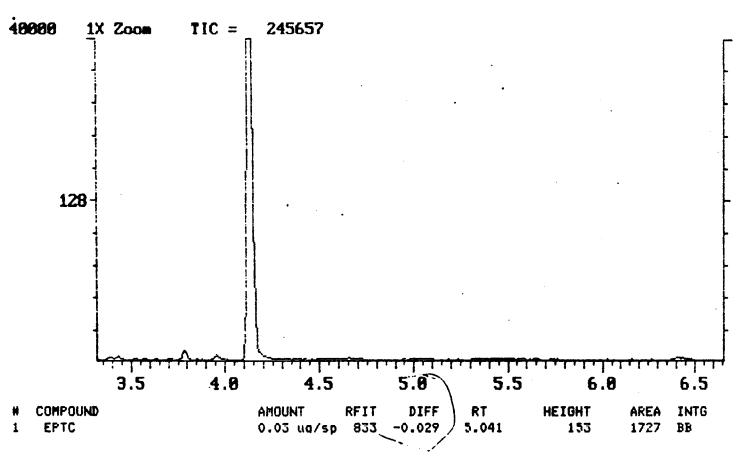
: ETAC BLANK EPTC USIS 128 ION Comment

Data file mLs sample uL injected 903-BLK 1.000 1.000 Acquired: 03 Sep 1997 4:34 pm

Calibration file : EPTC__XX Quantitation file: 903-BLK

15M DB-17 6169524B 10PSI SCOTT Column Chemist Instrument II S/N 036415420 1997 4:45 pm Instrument : Saturn Date printed : 03 Sep Plot Spooler screen Injections... Septum

136 48 , Insert/pre-column 48 , Column 1686



N.D.

ATTACHMENT J ARB Resin Sample Chromatogram - S1 - 2.92 ug/sample

: UHS 156 FRONT EPTC ANALYSIS Comment

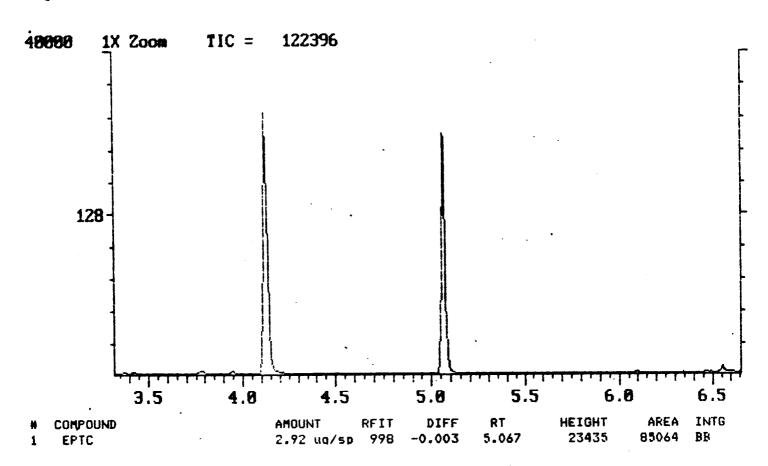
Acquired: 09 Sep 1997 1:13 am

908-156 3.000 2.000 Data file mLs sample uL injected

Quantitation file: 908-156 Calibration file : EPTC_XX

Column : 15M DB-Chemist : SCOTT Instrument : Saturn Date printed : 89 Sep Plot Spooler screen Injections... Septum DB-17 6169524B 10PSI II S/N 036415420 1997 1:24 am

187 179 , Insert/pre-column 179 , Column 1817



APPENDIX III QMOSB AUDIT REPORT



California Environmental Protection Agency



Air Resources Board

P.O. Box 2815 2020 L Street Sacramento, CA 95812-2815 http://www.arb.ca.gov



Peter M. Rooney Secretary for **Environmental** Protection

MEMORANDUM

TO:

George Lew, Chief

Engineering and Laboratory Branch

THROUGH:

Cook, Chief

(lit) Management and Operations

Support Branch

FROM:

Alice Westerinen, Manager Quality Assurance Section /

DATE:

December 5, 1997

SUBJECT:

FINAL EPTC 1996 QA SYSTEM AUDIT REPORT

Attached is the final quality assurance system audit report on the EPTC monitoring project conducted during October and November 1996, by the Engineering and Laboratory Branch of the Air Resources Board.

Thank you for participating in this audit. If you have any questions, please contact Mr. Trevor M. Anderson at (916) 323-0346.

Attachment

cc:

Trevor M. Anderson

Kevin Mongar

STATE OF CALIFORNIA AIR RESOURCES BOARD MONITORING AND LABORATORY DIVISION QUALITY ASSURANCE SECTION

SYSTEM AUDIT REPORT

AMBIENT MONITORING OF EPTC

IN

IMPERIAL COUNTY

FINAL

DECEMBER 1997

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EPTC MONITORING IN IMPERIAL COUNTY

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I. EXECUTIVE SUMMARY

In October and November 1996, the Engineering and Laboratory Branch (ELB) of the Air Resources Board (ARB) conducted a five week source impacted ambient air monitoring program for an application of EPTC to a field in Imperial County. This monitoring was conducted to determine if EPTC could be detected and measured in ambient air. The samples were collected and analyzed by ELB.

The Quality Assurance Section (QAS) of ARB's Monitoring and Laboratory Division (MLD) conducted a system audit of the field and laboratory operations to review the sample handling and storage procedures, analytical methodology, and method validation. In general, the laboratory practices were consistent with the Quality Assurance Plan for Pesticide Monitoring (ARB, February 4, 1994).

Additionally, QAS staff conducted performance audits of the air monitoring samplers. The performance audits of the air monitoring samplers were conducted to evaluate the flow rate accuracy. The flow rate audit was administered on September 6, 1996. The difference between the reported and assigned flow rates averaged -0.5% with a range of -4.8% to 0.5%.

QAS staff reviewed the sample storage stability study conducted by ELB, to determine the percent recovery of EPTC over time. The primary section of eight tubes was spiked with 2673 nanograms (ng) of EPTC. The spiked tubes were stored in the freezer at -20° Celsius and extracted/analyzed on storage days 0, 2, 6, and 29. Two tubes were analyzed on each day. The results of the stability study showed the EPTC samples had an average recovery rate of 101%, 110%, 110%, and 105% for days 0, 2, 6, and 29, respectively. No breakthrough occurred during the 24 hours of dynamic sampling at 2 liters per minute (LPM) air flow.

To determine the effectiveness of the analytical procedure, laboratory performance audits were conducted. In November 1996, a total of 20 QA audit samples were spiked with known amounts of QAS's standard solution of EPTC in ethyl acetate. These samples were submitted to ELB for analysis. The samples were prepared from EPTC standard solutions obtained from AccuStandard Inc.

The 20 audit samples were designated as quality assurance (QA) field spikes, QA trip spikes, and QA laboratory spikes. The QA field spikes were exposed to the same handling and storage conditions and also exposed to the same environmental and monitoring conditions as those occurring at the time of ambient sampling. The QA trip spikes followed the same handling and storage conditions as the ambient samples.

handling and storage conditions of the ambient samples. Finally, QA laboratory spikes were stored at ELB's storage freezer and then analyzed at the ELB laboratory.

The first set of ten QA spiked audit samples analyzed was QA laboratory spikes of EPTC in ethyl acetate. The QA spikes were stored in ELB's storage freezer at -20° Celsius for four days and were analyzed on November 20, 1996. The audit results for EPTC indicated a good recovery rate. The difference between the assigned and the reported total mass for EPTC laboratory spikes averaged 12.7% with a range of 5.4% to 23.4%. After review and discussion with ELB staff, the QA laboratory spiked audit data for EPTC were determined to be reasonable.

The next QA spiked audit samples analyzed were five QA trip spikes of EPTC in ethyl acetate. These samples were analyzed on November 22, 1996. The trip spiked audit results for EPTC indicate a difference between the assigned and the reported total mass averaged 19.1% with a range of 17.6% to 20.6%. After review and discussion with ELB staff, the QA trip spiked audit data for EPTC were determined to be reasonable.

The five QA field spiked audit samples analyzed for EPTC in ethyl acetate were conducted on November 22, 1996. The QA field spiked audit results for EPTC indicate a difference between the assigned and the reported total mass average of 10.4% with a range of 8.1% to 13.1%. After review and discussion with ELB staff, the QA field spiked audit data for EPTC were determined to be reasonable.

Blanks were assigned for each batch of the EPTC QA laboratory, trip, and field spiked samples. No contamination of the blanks was detected.

QAS staff reviewed the "head-to-head" analyses between the QA standard solution of EPTC in ethyl acetate and ELB's working standard, conducted by ELB. The analyses were conducted on November 5 and 6, 1996. ELB's laboratory standard concentration was created by using a pure or "neat" solution of EPTC, while QA's standard solution of EPTC was procured by AccuStandards Inc. In this comparison, ELB staff found the ELB's EPTC laboratory standard to be 25.1% and 21.6% different than the QA standard solution. The elevated difference between QA's and ELB's solutions is responsible for the higher recovery rates of the QA spiked samples. After review and discussion with ELB staff, the QA standard solution for EPTC was determined to be reasonable.

II. CONCLUSION

Operations

The records for field operations, sample handling procedures, analytical methodology, and method validation were in agreement with the Quality Assurance Plan for Pesticide Monitoring.

Field Flow Rates

The results of the reported flow rates were in agreement with the actual flow rates measured by QAS staff.

Laboratory Accuracy

The QAS audit results for EPTC laboratory, trip, and field spikes resulted in good recovery levels. The difference between the assigned and the reported total mass for EPTC laboratory spikes averaged 12.7% with a range of 5.4% to 23.4%. The QA trip spiked audit results for EPTC indicate a difference between the assigned and the reported total mass averaged 19.1% with a range of 17.6% to 20.6%. The QA field spiked audit results for EPTC indicate a difference between the assigned and the reported total mass average of 10.4% with a range of 8.1% to 13.1%. Blanks were assigned for each batch of the EPTC QA laboratory, trip, and field spiked samples. No contamination of the blanks was detected. After review and discussion with ELB staff, the QA laboratory, trip, and field spike audit data for EPTC were determined to be reasonable.

QAS staff reviewed the "head-to-head" analyses between the QA standard solution of EPTC in ethyl acetate and ELB's working standard, conducted by ELB. The analyses were conducted on November 5 and 6, 1996. ELB's laboratory standard concentration was created by using a pure or "neat" solution of EPTC, while QA's standard solution of EPTC was procured by AccuStandards Inc. In this comparison ELB staff found the ELB's EPTC laboratory standard to be 25.1% and 21.6% different than the QA standard solution. The elevated difference between QA and ELB's solutions is responsible for the higher recovery rates of the QA spiked samples. After review and discussion with ELB staff, the QA standard solution for EPTC was determined to be reasonable.

Impact on Data

After reviewing QAS spiking standard solution handling, storage, and shipping records, along with records for analyses of QA spikes at ELB's laboratory, concentration for the standard solutions, stability studies, and all other

laboratory and field procedures, it has been determined that QAS analytical performance audit data for EPTC produced good recovery rates of the QAS spiking solution.

III. RECOMMENDATIONS

- 1. Before handling and assembling the spiking solution and samples, laboratory procedures and practices should be thoroughly reviewed and followed by all parties involved.
- 2. The monitoring protocol for a pesticide should be finalized before actual sampling begins.

IV. INTRODUCTION

In October and November 1996, the Engineering and Laboratory Branch (ELB) of the Air Resources Board (ARB) conducted a five-week source impacted ambient air monitoring program for an application of EPTC to a field in Imperial County. This monitoring was conducted to determine if EPTC could be detected and measured in ambient air. The samples were collected and analyzed by ELB. The ARB's Monitoring and Laboratory Division's (MLD) Quality Assurance Section (QAS) staff conducted a system audit of the field and laboratory operations. Performance audits of the air samplers' flow rates and of the analytical method were also conducted.

V. AUDIT OBJECTIVE

The system audit was conducted to determine whether the quality control practices for the handling and storage of samples, analytical methodology, and method validation were consistent with the Quality Assurance Plan for Pesticide Monitoring (ARB, February 4, 1994). Performance audits were conducted to evaluate the accuracy of the air samplers' flow rates and the analytical method.

VI. FIELD AND LABORATORY OPERATIONS

A system audit of the field and laboratory operations was initiated in August 1996, through a questionnaire submitted to ELB staff. Additionally, the "Protocol for the Ambient Air Monitoring of EPTC in Imperial County During Fall, 1996" and ELB's "Standard Operating Procedure for the Analysis of EPTC in Ambient Air" were reviewed by QAS staff. In general, the laboratory practices were consistent with the Quality Assurance Plan for Pesticide Monitoring (ARB, February 4, 1994).

Ambient Air Sampling, Sample Handling and Storage

Samples were collected by drawing ambient air at a measured rate through sample tubes containing XAD-2 resin. Once sampled, the exposed XAD-2 resin tubes were stored either on dry-ice or in a freezer until desorbed with 3 milliliters (mL) of ethyl acetate in the laboratory. The flow rate was accurately measured and the sampling system operated continuously at the exact operating interval. The resin tube was protected from direct sunlight using a rain shield and was supported 1.5 meters above ground during the sampling period. An air sampler consisted of the glass cartridge containing XAD-2, connected with Teflon tubing to an in-line rotameter, which in turn was connected to an air pump. A sketch of the sampling apparatus is shown in Attachment 1.

The samplers' rotameters were set to an indicated flow rate of 2.0 LPM. The sampling was conducted following the schedule specified in the sampling protocol. The samples were removed from the sample train, capped, and identification labels were affixed to each tube. Each sample was placed in a culture tube. Up to five culture tubes were placed in a zip-lock plastic bag. The samples were stored on dry ice and held in the field for up to one week prior to shipment to the laboratory. Upon receipt at ELB laboratory in Sacramento, the samples were either analyzed immediately or stored in a freezer until extraction and analyses were conducted. All samples were analyzed within two weeks of receipt by ELB.

Sample Analysis

The analytical method used was developed by ELB and described in the "Standard Operating Procedure for the Analysis of EPTC in Ambient Air." The method calls for the XAD-2 resin to be stored in a refrigerator or ice chest until desorbed with 3 mL of ethyl acetate. The sample is desorbed by pouring the XAD-2 resin into a vial and adding 3 mL of ethyl acetate. The sample is then placed on a desorption shaker (or ultra sonic water bath) for 30 minutes. After being removed from the shaker, the solvent is removed from the XAD resin and is stored in a second vial at -20° Celsius until analysis. 2.0 microliter (uL) sample is then injected into the gas chromatograph (GC) and analyzed. The injected samples were analyzed on a Hewlett Packard model 5890 gas chromatograph with a DB-35 capillary column and a mass selective detector Five concentrations of EPTC (using triplicate injections per level) were used to establish the initial instrument standard calibration curve at 111 ng/mL, 223 ng/mL, 446 ng/mL, 891 ng/mL, and 1,782 ng/mL.

The following quality control activities were performed to monitor and document the quality of the data: trip blanks were submitted with every batch delivered from the field and about 10% of the samples were analyzed in replicate to document analytical precision; precision checks of the data showed less than ±10% difference; field duplicates from collocated sites were collected once per week at each site; all of the samples were analyzed by Gas Chromatograph Mass Spectroscopy Selective Ion monitoring to confirm the identity of the analyte.

Method Validation

The limit of detection (LOD) criteria was determined by using multiple determinations of low concentrations of EPTC. The LOD was calculated to be 10.6 ng/mL for EPTC. Collection and extraction efficiency was determined by using 7.5 uL and 15 uL spiked samples. The percent recovery for EPTC was 101% for the 7.5 uL spiked samples and 90.3% for the 15.0 uL spiked samples.

A sample storage stability study was conducted by ELB to determine the percent recovery of EPTC over time. The primary section of eight tubes was spiked with 2673 ng of EPTC. The spiked tubes were stored in the freezer at -20° Celsius and extracted/analyzed on storage days 0, 2, 6, and 29. Two tubes were analyzed on each day. The results of the stability study shows the EPTC samples had an average recovery rate of 101%, 110%, 110%, and 105% for days 0, 2, 6, and 29, respectively. No breakthrough occurred during the 24 hours of dynamic sampling at 2 LPM air flow.

Documentation

All the samples received at the laboratory were accompanied by chain-of-custody records. Field data sheets containing the sample collection information were retained by ELB. The information recorded in the field data sheets included sampler ID, sampling date, start and stop times, flow rate, and comments about unusual conditions.

Laboratory and instrument maintenance logs were kept in bound notebooks with numbered pages. The entries made in the laboratory book included sample number, sample type, the date sample was received, collection date, date of analysis, results of analysis, and analyst. The raw analytical data were recorded on electronic files and will be kept up to six years by ELB.

VII. PERFORMANCE AUDITS

It should be noted that the percent difference for all Tables is calculated by using the following equation:

Reported Value - True Value x 100
True Value

Flow Rate Audit

The flow rate for each sampler used was audited on September 6, 1996, following the procedures outlined in Attachment 2. The audit was conducted with a 0 to 3 LPM mass flow meter traceable to the National Institute of Standards and Technology (NIST). The difference between the reported and true flow rates for the ambient air samplers averaged -0.5% and ranged from -4.8% to 0.5% (Table 1).

Table 1

Results of the Flow Audit Conducted on the Ambient Samplers Used During the Monitoring for EPTC

Sampler Number	Reported Flow (LPM)	True Flow (LPM)	Percent Difference
1A	1.88	1.88	0.0
1B	1.88	1.88	0.0
2A	1.88	1.79	-4.8
2B	1.88	1.89	0.5
3A	1.88	1.88	0.0
3B	1.88	1.89	0.5
4A	1.88	1.88	0.0
4B	1.88	1.88	0.0

Analytical Performance Audit

In October and November 1996, a total of 20 QA ambient audit samples were spiked with known amounts of QAS's standard solution of EPTC in ethyl acetate, following the procedures outlined in Attachment 3. The 20 QA audit samples were designated as QA field spikes (5), QA trip spikes (5), and QA laboratory spikes (10). The QA field spikes were exposed to the same handling and storage conditions and were also exposed to the same environmental and monitoring conditions as those occurring at the time of ambient sampling. The QA trip spikes followed the same handling and storage conditions of the ambient samples.

The ten QA laboratory spikes were stored at ELB's storage freezer at -20° Celsius for four days before extraction and analysis. The QA laboratory spikes were analyzed by ELB on November 20, 1996. The audit results for EPTC indicated a good recovery rate. The difference between the assigned and the reported total mass for EPTC laboratory spikes averaged 12.7% with a range of 5.4% to 23.4% (Table 2). Two blanks were assigned with the spike samples and no contamination of the blanks was detected. After review and discussion with ELB staff, the QA laboratory spike audit data for EPTC were determined to be reasonable.

Table 2

Results of Analyses of the QA Laboratory

Spikes for EPTC

Sample ID	Assigned Mass EPTC (ng)	Reported Mass EPTC (ng)	Percent Difference
QA-EPTC-L1A	0.00	0.00	0.0%
QA-EPTC-L2A	191.25	230.00	20.3%
QA-EPTC-L3A	191.25	236.00	23.4%
QA-EPTC-L4A	688.50	761.00	10.5%
QA-EPTC-L5A	688.50	726.00	5.4%
QA-EPTC-L6A	0.00	0.00	0.0%
QA-EPTC-L7A	1,147.50	1,270.00	10.7%
QA-EPTC-L8A	1,147.50	1,250.00	8.9%
QA-EPTC-L9A	382.50	430.00	12.4%
QA-EPTC-L10A	A 382.50	420.00	9.8%

The five QA trip spiked audit samples were exposed to the same handling and storage conditions as those occurring at the time of ambient monitoring. The trip spikes were shipped, in an ice chest containing dry ice, from ELB laboratory to the Imperial County ambient air monitoring station. At the Imperial site, the trip spikes were stored for four days in an ice chest containing dry ice, packaged with QA field spikes, and returned to ELB laboratory for analysis.

The QA trip spiked audit samples were analyzed on November 22, 1996. The trip spike audit results for EPTC indicates a difference between the assigned and the reported total mass averaged 19.1% with a range of 17.6% to 20.6% (Table 3). One blank was assigned with the spike samples and no contamination of the blank was detected. After review and discussion with ELB staff, the QA trip spike audit data for EPTC were determined to be reasonable.

Table 3

Results of Analyses of the QA Trip
Spikes for EPTC

Sample ID	Assigned Mass EPTC (ng)	Reported Mass EPTC (ng)	Percent Difference
=======================================			
QA-EPTC-T1A	1,147.50	1,370.00	19.4%
QA-EPTC-T2A	1,147.50	1,350.00	17.6%
QA-EPTC-T3A	688.50	818.00	18.8%
QA-EPTC-T4A	688.50	830.00	20.6%
OA-EPTC-T5A	0.00	0.00	0.0%

The five QA field spikes were installed into the pesticide air monitor at this station and exposed to 24 hours of ambient air sampling thru-the-tube samples at a rate of 2 LPM. A replicate air sampler (collocated) was used to collect and determine the background ambient air concentrations. After exposure to the field conditions, the samples were packaged, stored, and shipped in an ice chest containing dry ice to ELB for analysis.

The five QA field spikes audit samples analyzed for EPTC were conducted on November 22, 1996. The QA field spike audit results for EPTC indicates a difference between the assigned and the reported total mass average of 10.4% with a range of 8.1% to 13.1% (Table 4). One blank was assigned with the spike samples and no contamination of the blank was detected. After review and discussion with ELB staff, the QA field spike audit data for EPTC were determined to be reasonable.

Table 4

Results of Analyses of the QA Field
Spikes for EPTC

Sample ID	Assigned Mass EPTC (ng)	Reported Mass EPTC (ng)	Percent Difference
=========	============		
QA-EPTC-F1A	0.00	0.00	0.0%
QA-EPTC-F2A	688.50	744.00	8.1%
QA-EPTC-F3A	688.50	779.00	13.1%
QA-EPTC-F4A	994.50	1,100.00	10.6%
OA-EPTC-F5A	994.50	1,090.00	9.6%

The QAS analytical performance audit information for laboratory, trip, and field spikes of EPTC was conducted by reviewing QA spiking standard solution handling, storage, and shipping records, along with records for analyses of QA

spikes at ELB's laboratory. The following are the results of the review.

The QAS's EPTC standard solutions were procured by AccuStandard Inc. The standards had an expiration date of November 1997. No spiking or calculation errors were found when reviewing QA spiking logbook.

The stability studies conducted by ELB staff determined that EPTC was stable for up to 29 days when stored at -20° Celsius. The QA laboratory, trip, and field spiked samples were transported, stored and analyzed within the 29 day stability requirement. No thermometer or recording of the temperature was logged during the storage of the spiked samples.

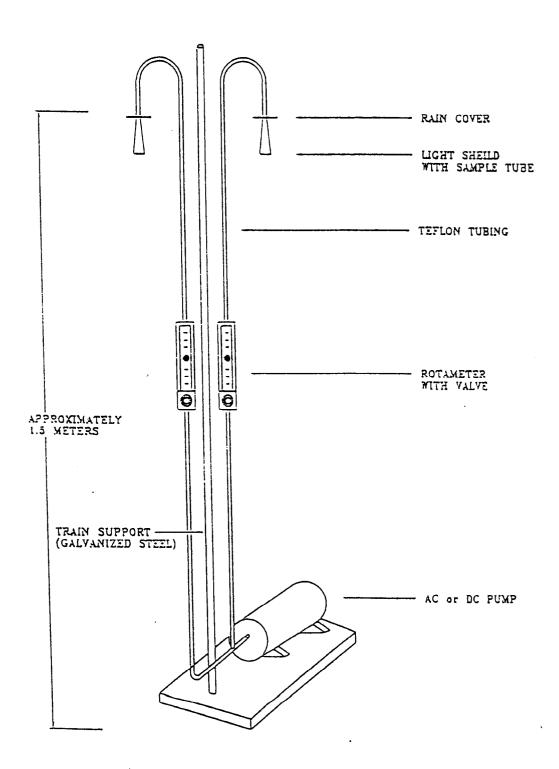
The Hewlett Packard 5890 GC was calibrated daily during the analyses of the ambient samples and QA spiked samples.

Review of the chromatograms and the sample analyses data showed no data transfer or calculation errors. About 10% of the samples were analyzed in replicate to document analytical precision.

QAS staff reviewed the "head-to-head" analyses between the QA standard solution of EPTC in ethyl acetate and ELB's working standard, conducted by ELB. The analyses were conducted on November 5 and 6, 1996. ELB's laboratory standard concentration was created by using a pure or "neat" solution of EPTC, while QA's standard solution of EPTC was procured by AccuStandards Inc. In this comparison ELB staff found the ELB's EPTC laboratory standard to be 25.1% and 21.6% different than the QA standard solution. The elevated difference between QA and ELB's solutions is responsible for the higher recovery rates of the QA spiked samples.

Based on the information provided, it has been determined that QAS analytical performance audit data for EPTC produced good recovery rates of the QAS spiking solution and the impact on the ambient data compared with QAS spiking solutions for EPTC were found to be reasonable.

AIR SAMPLER USED IN MONITORING OF EPTC



FLOW RATE AUDIT PROCEDURES FOR AIR SAMPLERS USED IN PESTICIDE MONITORING

Introduction

Air samplers are audited using a calibrated differential pressure gauge or a mass flow meter that is standardized against a National Institute of Standards and Technology (NIST) traceable flow calibrator. The audit device is connected in series with the sampler's flow meter. The flow rate is measured while the sampler is operating under normal sampling conditions. The sampler's indicated flow rate is corrected based on its calibration, and the true flow is calculated from the audit device's calibration curve. The sampler's reported flow is compared to the true flow, and a percent difference is determined.

Equipment

The basic equipment required for the air sampler flow audit is listed below. Additional equipment may be required depending on the particular configuration and type of sampler.

- 1. NIST-traceable mass flow meter.
- 2. Calibrated differential pressure gauge with laminar flow element.
- 1/4" outer diameter Teflon tubing.
- 4. 1/4", stainless steel, Swagelock fittings.

Audit Procedures

- 1. If power is available, connect the mass flow meter into a 110 VAC outlet, and allow it to warm up for at least ten minutes. Otherwise, perform the audit with the calibrated differential pressure gauge.
- Connect the inlet port of the audit device to the outlet port of the sampler's flow control valve with a five-foot section of Teflon tubing and Swagelock fittings.
- 3. Connect the outlet port of the audit device to the pump with another five-foot section of Teflon tubing and Swagelock fittings.
- 4. Allow the flow to stabilize for at least one to two minutes and record the flow rate indicated by the sampler and audit device's response.

5. Calculate the true flow rate from the audit device's response and record the results. Obtain the corrected sampler flow rate from the field operator. Calculate the percent difference between the true flow rate and the reported flow rate.

The percent difference is calculated by using the following equation:

Reported Flow - True Flow x 100
True Flow

PERFORMANCE AUDIT PROCEDURES FOR THE LABORATORY ANALYSIS OF EPTC

Introduction

The purpose of the laboratory performance audit is to assess the accuracy of the analytical method used by the laboratory to measure the ambient concentrations of EPTC. The audit is conducted by submitting audit samples spiked with known concentrations of EPTC in ethyl acetate. The analytical laboratory reports the results to the Quality Assurance Section. The difference between the reported and the assigned concentrations is used as an indicator of the accuracy of the analytical method.

Materials

- 1. EPTC, 76.5 μ g/mL EPTC in ethyl acetate, AccuStandard Inc., Lot #A6100358, Expires 11/1/97.
- XAD-2 adsorbent resin tubes, supplied by SKC West Inc.

Safety Precautions

Prior to handling any chemical, read the manufacturer's Material Safety Data Sheets. Avoid direct physical contact with chemicals. Avoid breathing vapors. Use only under a fume hood. Wear rubber gloves, safety glasses, and protective clothing.

Preparation of Audit Samples

Prepare five field samples, five trip samples, and ten laboratory audit samples by spiking the XAD-2 adsorbent cartridges with the volume of EPTC spiking solution indicated in Table 1 below. Using a microsyringe, insert the needle into the primary section of the XAD-2 cartridge, and push the plunger slowly while spiking the XAD-2 adsorbent resin.

Table 1

Volume of EPTC in Ethyl Acetate Used to Spike the QA Ambient Audit Samples

Sample	EPTC Spiking								
ID	Solution Volume (uL)								
Field Spikes (AccuStandard S	Field Spikes (AccuStandard Standard Solution)								
QA-EPTC-F1A	0.0								
QA-EPTC-F2A	9.0								
QA-EPTC-F3A	9.0								
QA-EPTC-F4A	13.0								
QA-EPTC-F5A	13.0								
Trip Spikes (AccuStandard St	candard Solution)								
QA-EPTC-T1A	15.0								
QA-EPTC-T2A	15.0								
QA-EPTC-T3A	9.0								
QA-EPTC-T4A	9.0								
QA-EPTC-T5A	0.0								
Laboratory Spikes (AccuStance	dard Standard_Solution)								
QA-EPTC-L1A	0.0								
QA-EPTC-L2A	2.5								
QA-EPTC-L3A	2.5								
QA-EPTC-L4A	9.0								
QA-EPTC-L5A	9.0								
QA-EPTC-L6A	0.0								
QA-EPTC-L7A	15.0								
QA-EPTC-L8A	15.0								
QA-EPTC-L9A	5.0								
QA-EPTC-L10A	5.0								

APPENDIX IV PCA'S APPLICATION RECOMMENDATION and REPORT

WA003

Western Farm Service, Inc. PESTACIPERNSEARECEMBENDETION Multiple Field Rec. 3348 CLAUS ROAD MODESTO, CA 95355 Expires: (209) 551-1424 Completed: Chop: CORN/FIELD Grower: AHLEM FARMS 9072 COLUMBUS Acea: 205 Acres Del Tkt#: HILMAR CA 95324 7U#: 209 634-2762 ACA: HENRY SOARES 2804 Acct#: 5001491 Pest. Permit#: 24-97-2405483 Applr: ENHIRONME Fldmn: HENRY SOARES 2804 County Site Section Township BAM Field Range Treatment Area C924 6S -9E -188 Acres S/W AMERICAN & FAITH HOME 13-M MA 001 201 S/W BLOSS & FAITH HOME **CR24** 24-6S -39 Acres **J28641** 992 6S -CR24 23-N/W MITCHELL & WILLIAMS 10E -**#1003** 883 39 Acres Recommendation#: 5202 Proposed Treatment: 178 Acres Material REG. # RATE /100 gal Band Mat. Req. Target Pest ERADICANE 6.7-E 18182-00223-00-00000 7.68 pts /Treated Ac 28.00 No 155.75 gal PURPLE NUTSEDGE 10182-00018-00-00000 12.00 exs /Treated Ac 48.00 No 16.69 gal CUTHORNS AMBUSH INSECTICISE Gallons of Diluent/Treated Acre: 25 ----- SPECIAL INSTRUCTIONS ------ERADICANE 6.7-E: DO NOT USE IN THE 10 SOUTHERNMOST COUNTIES. AMBUSH: APPLY FROM 5 DAYS PRIOR TO PLANTING UP TO CROP EMERGENCE. *Restricted: YES, CERTIFIED APPLR ONLY Days to Harvest: N/A Permit Required: NO Notice of Intent Required: NO Avoid Drift: YES Avoid Water Contamination: YES ¿Toxic to Bees: YES * Chemical Category: II WARNING Closed Mixing System Required: NO Toxic to Fish: YES Posting Required: NO Toxic to Birds: NO Non Re-entry Interval: 12.00 HOURS Feed/Graze Treated Area/Crop於YEE階級級 Plantback Restriction: 2 Months *NOTIFY BEEKEPERS AT LEAST 48 HRS BEFORE APPLICATION Lays to Pasture: 0 ATEPO, KERN AND SAN BERNARDINO COUNTIES. DO NOT USE IN OR SOUTH OF SAN EUE CRITERIA / ENVIRONMENTAL CHANGES (1982) 1. PESTS AT ECONOMIC LEVELS SHIP **852197** DEL TO SOUTH WEST CORNER OF FAITH-HONE & AMERICAN PUT $\S H_{\mathcal{S}}$ OFFICE The execution of this recommendation certifies that alternative and mitigation measures that would substantially lessen any significant adverse ispact on the environment have been considered and, if feasible, adopted. HENRY, SOARES 8804 Signatures I Grower Signature:

> Copyright 1984-1996 CDMS, Inc. SE# AN0344 =READ THE LABEL= *** RECOMMENDATION CONTINUED ON NEXT PAGE ***

Post-it Fax Note 7671	Date 12/30 pages 1
TO KEVIN MONGER	From DAN CISMOUSKI
ARB	Ca. MERCEO C.A.C.
Phone #	Phone 9 385-7421
Fax 1(916) 263-2067	Fex (204) 725-3536

Submit to AGRICULTURAL COMMISSIONER within 7 days of application

2921

(1) CAC

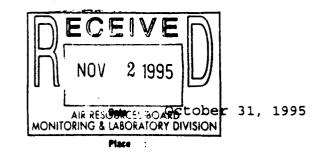
33-025 (REV. 3/90)

APPENDIX V DPR'S MONITORING RECOMMENDATION

State of California

Memorandum

George Lew, Chief
Engineering and Laboratory Branch
Monitoring and Laboratory Division
Air Resources Board
600 North Market Boulevard
Sacramento, California 95812



From Department of Pesticide Regulation

1020 N Street, Room 161
Sacramento, California 95814-5624

Subject . MONITORING RECOMMENDATION FOR EPTC.

Attached is the Department of Pesticide Regulation's recommendation for monitoring the herbicide EPTC. This recommendation is provided pursuant to the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5). As you know, monitoring recommendations are made using historical use information for the pesticide in question. For this reason, it is essential that the agricultural commissioner, in the county or counties where monitoring will be conducted, be consulted prior to the onset of air monitoring.

We anticipate submission of air monitoring data by April 1997.

If you have any questions please contact Kevin Kelley, of my staff, at (916) 324-4187.

John S. Sanders, Chief Environmental Monitoring

and Pest Management Branch

(916) 324-4100

attachment

CC: Paul H. Gosselin, DPR
Charles M. Andrews, DPR
Ronald J. Oshima, DPR
Gary Patterson, DPR
Barry Cortez, DPR
John Donahue, DPR

Kevin Kelley, DPR Madeline Brattesani, DPR Genevieve Shiroma, ARB Don Fitzell, ARB Cara Roderick, ARB

Steven L. Birdsall, Agricultural Commissioner Imperial County Michael J. Tanner, Agricultural Commissioner Merced County Erwin B. Eby, Agricultural Commissioner San Joaquin County



State of California
Department of Pesticide Regulation
1020 N Street
Sacramento, California 95814-5624

Staff Report

USE INFORMATION AND AIR MONITORING RECOMMENDATION FOR THE PESTICIDAL ACTIVE INGREDIENT EPTC

October 1995

Principal Author

Kevin C. Kelley Associate Environmental Research Scientist

MONITORING RECOMMENDATION FOR EPTC

BACKGROUND

In order to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5), the Department of Pesticide Regulation (DPR) has previously requested that the Air Resources Board (ARB) document the airborne concentrations of the pesticide EPTC (Dipropylcarbamothioc acid S-ethyl ester). This recommendation provides background and recent use information on EPTC-containing products, and identifies how they are used.

EPTC (CAS: 759-94-4) is a colorless to light yellow liquid with an amine-like odor. Technical grades are yellow. EPTC has a molecular formula of C₉H₁₉NOS, a formula weight of 189.32 g/mole, and a specific density of 0.960 at 25 °C. It has a water solubility of 375 mg/L at 25 °C, a Henry's Constant of 1.0 x 10⁻⁵ atm·m³/mol at 20–25 °C, and a vapor pressure of 3.4 x 10⁻² mmHg at 20 °C. EPTC is miscible with most organic solvents.

EPTC is rapidly metabolized by soil micro-organisms to carbon dioxide, mercaptan, and amino residues. Mineralization has not been reported in sterile soils due to the lack of production of carbon dioxide. Soil half-life $(t_{1/2})$ ranges from 4-6 weeks when applied at recommended rates. In plants, EPTC is rapidly metabolized to carbon dioxide and other naturally occurring plant constituents. EPTC sulfoxide has been reported in some soils and in corn plants.

The acute oral LD₅₀ of EPTC for male rate and mice is 1,700 and 3,200 mg/kg. The LC₅₀ (48 hour) for rainbow trout is 19 mg/L, and 27 mg/L for bluegill sunfish. EPTC has entered the risk assessment process at DPR under the SB 950 (Birth Defect Prevention Act of 1984) based on its potential neurotoxicity, mutagenicity, and teratologic and chronic toxicity adverse health effects.

USE OF EPTC

As of October 5, 1995, there were 11 active registrations for products containing EPTC, all of which are agricultural products. EPTC is a pre-plant/pre-emergent herbicide for the control of annual and perennial grasses and broadleaf weeds. EPTC may also be top-dressed onto corn following germination, or applied through irrigation systems. EPTC is formulated as either a granular or as an emulsifiable concentrate. The Signal Word found on these EPTC-containing products is "Caution".

Use of EPTC for 1993, 1992 and 1991 is summarized in the following tables: Table 1, shows EPTC use by year; and Table 2, shows EPTC applications in Imperial and San Joaquin Counties. Agricultural use of EPTC for the twelve counties listed in Table 1, accounts for 85% to 88% of total EPTC use. The remaining 12% to 15% of the total use is applied agriculturally (in counties not listed in table 1), or used as potato seed treatments (approximately 1% of total use). Less than 0.025% of the total amount of EPTC used was for non-agricultural purposes (landscape maintenance, rights-of-way).

Table 1. EPTC Use by Year (Pounds of Active Ingredient)

County	1993	1992	1991
Fresno	34,048.3	31,380.4	18,144.0
Glenn	23,764.3	23,070.2	22,096.7
Imperial	143,652.5	186,044.7	259,597.8
Kern	63,027.0	64,320.9	65,892.2
Kings .	36,960.4	13,234.4	12,718.7
Merced	43,682.9	45,076.2	45,354.8
Riverside	29,711.5	38,098.6	37,727.6
Sacramento	38,430.7	26,781.3	19,104.5
San Joaquin	92,001.6	66,994.6	74,975.9
Stanislaus	47,395.6	47,364.2	56,660.8
Tulare	21,100.9	24,558.6	21,172.3
Yolo	32,945.3	20,975.0	25,650.1
County Tòtals	606,721.0	589,899.1	659,095.4
CALIFORNIA TOTAL	713,561.9	667,274.5	751,977.1

The PUR data summarized in Table 1 show that Imperial and San Joaquin Counties routinely receive the greatest applications of EPTC. Table 2, summarizes the total amounts and rates of EPTC applied in these counties during the months of greatest use. Yearly applications are largest in Imperial County followed by yearly applications in San Joaquin County. However, applications in Imperial County occur throughout the year while applications in San Joaquin County occur during a spring window of April, May and June. Applications of EPTC during the highest season of use for Imperial and San Joaquin Counties are shown in Table 2.

Table 2. EPTC applications in Imperial and San Joaquin Counties (Pounds of Active Ingredient)

Imperial County	1993	1992	1991	
October (lbs AI)	29,601.5	26,019.6	19,309.2	
(Rate)	2.06	2.41	2.12	
November (lbs AI)	19,603.6	35,734.1	33794.4	
(Rate)	2.22	3.10	2.29	
San Joaquin County	1993	1992	1991	
April (lbs AI)	23,998.5	25,337.8	22,628.6	
(Rate)	4.29	4.38	5.13	
May (lbs AI)	36,791.0	18,966.5	22,699.8	
(Rate)	5.03	3.59	4.25	

EPTC is used for pre-plant and pre-emergence control of annual and perennial grasses and broadleaf weeds. In corn, EPTC is used for the eradication of barnyardgrass and nutsedge, and for suppression of johnsongrass. If applied as a granule or spray, immediate incorporation into soil is necessary or herbicidal properties are lost. EPTC may also be applied through sprinkler irrigation systems. Planting should occur as soon as possible following application and always within 2 weeks of application.

RECOMMENDATIONS

Ambient Air Monitoring.

The use patterns for EPTC suggest that monitoring should occur over a 30- to 45-day sampling period in either Imperial or San Joaquin County. Sampling may be conducted during the months of October and November in Imperial County; alternatively, sampling may be conducted in San Joaquin County during April and May. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. In Imperial County, sampling sites should be located near alfalfa and sugarbeet growing areas. In San Joaquin County, sampling sites should be located near corn growing areas. Ambient samples should not be collected from samplers immediately adjacent to fields where EPTC is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during

the sampling period. Background samples should be collected in an area distant to EPTC applications.

Replicate (co-located) samples are needed for five dates at each sampling location. Two co-located samplers (in addition to the primary sampler) should be run on those days. The date chosen for replicate samples should be distributed over the entire sampling period. They may, but need not be, the same dates at every site. Field blank and spike samples should be collected at the same environmental (temperature, humidity, exposure to sunlight) and experimental (air flow rates) conditions as those occurring at the time of ambient sampling.

Monitoring of an Application Site.

The use patterns for EPTC suggest that application-site monitoring should be collected during the months of April, May or June, and that monitoring should be associated with applications to corn. Application rates to corn generally range from 4.5 to 6 lbs AI/acre in San Joaquin and Merced Counties. Monitoring may be conducted in San Joaquin County during April or May, or in Merced County during May or June. EPTC is extensively applied during these periods so care should be taken so that nearby applications do not contaminate collected samples. A three day monitoring period should be established with sampling times as follows. Application + 1 hour, followed by one 2-hour sample, one 4-hour sample, two 8-hour samples and two 24-hour samples. A minimum of four samplers should be positioned, one on each side of the field. A fifth sampler should be co-located at one position. Since EPTC is extensively used in the area, background samples should collect enough volume (either 12 hours at 15 liters/min., or a shorter period with a higher volume pump) to permit a reasonable minimum detection level. Ideally, samplers should be placed a minimum of 20 meters from the field. Field blank and field spike samples should be collected at the same environmental (temperature humidity, exposure to sunlight) and experimental (similar air flow rates) conditions as those occurring at the time of sampling.

We also request that you provide in the monitoring report: 1) An accurate record of the positions of the monitoring equipment with respect to the field, 2) an accurate drawing of the monitoring site showing the precise location of the meteorological equipment, trees, buildings, etc., 3) meteorological data collected at a minimum of 15 minute intervals including wind speed and direction, humidity, and comments regarding degree of cloud cover, and 4) the elevation of each sampling station with respect to the field, and the orientation of the field with respect to North (identified as either true or magnetic North).

APPENDIX VI

APPLICATION AND AMBIENT FIELD LOG SHEETS

Project: EPTC Application Project #: C97-021

	Log Number	Sample ID	Date	Time	Comments		ast ly cloudy taken by
	1	SB	5/28 97	1240	Sample: on A- notice	K	KEM
	2	5 F51	5/2:/47	1240	Spikes on A- who		
.	3	WE	5/21/17	1245	'		
	4	WFS2	5/26/17	1245			
	5	NE	5/26/67	1248			
	6	NF53	5/26/97	1248			
	7	ER	5/26/97	1250			
	8	E FS4	5/2/197	1250			
	9	5 /	5/20/47	06 56 1555			
	10	5/1	5/27/97	0650			
*	//	WI	5/27/17	1500			
*	12	NI	5/27/47	1665			
*	13	El	5/27/47	0705			
	14	52	5/27/27	1555			
	15	520	5/20/17	1515			
* 7	16	WZ	5/27/47	1800			
*	17	N2	5/27/45	1605	·		
*	18	<i>I</i> 2	5/27/17	1605			V
	19	53	5/27/47 5/20/47	1755			
	20	531)	5/27/47	1755			
	21	w3	5/27/17 5/24/17	1800 2200			
	22	N3	5/27/47	2205	switchesthous to 10A & 10R		
•	23	E3	5/27/a7 5/27/ar	1325		· ·	V

Project: EPTC Application Project #: C97-021

	Log Number	Sample ID	Date	Time	Comments	weather o = overca pc = partly k = clear	cloudy
	24	54	5/28	2200		Suck	KIM
	25	546	5,28,	2:20			/
	26	W4	5/28	5705			
	スク	N4	5/27/97	2205			
	28	F 4	5/27/47	2205		N.	1
_	29	57	5/29 5/24	0700		K	KEM
-	30	55L	5/28	0700			
1	3 ;	W5	5/25 5/20	0700			
1	32	NS	5/27	0705			
-	33	E5	5/28 5/20	0710			4
†	34	56	5/35	0300			
4	35	560	5/3A 5/30	0800			
*	36	.W6	5/30	0910			
¥	37	N6	5/22 5/30	0110			
*	38.	£6	5/21	0715			
	39	751	5/30	0815			
	40	T52	5/30	0815			
	41	753	5/30	0815			
	42	754	5/50				
	43	B	5/30	0815			
				<u> </u>			

Project: EPTC Ambient Project #: C96-035

Log Number	Sample ID	Date	Time	Comments	o = o pc =	weather o = overca pc = parti k = clear		ly cloudy	
١	ARB-IA			ARB Calexico	K		KEL	<u></u>	
7	ARB-N3	10-9	1000						
3	H-IA	10-9	0945	Heber Fire Napartment				_	
4	H-1B	10-9	0905						
5	MV-)A	10-9	0920	meadons Union school					
6	MV-1B	10-9	0920	0.0400000					
7	EL-IA	10-10	0955	EL (en 40					
8	EL-1B	10-9	A55 MID						
9	EL-IL	10-9	0955						
10	EL-10	10-9	1955 1910						
11	IH-IA	10-9	1010 10830	Imperial High					
	IH-18	10-9	1010	I when the strings		1			
13	エルーン	10-10	0430						
14	EL-21	10-10	0910			1			
 	el-lb	10-10	0910			1			
16	MV-X	10-10	0780						
17	14-5	10-10	0740			†		 	
18	17 ° &	1040	1000			+			
19	B-5	10-11	0800	a. k		\dagger			
	0-7	10-11	0825	Blank	<u> </u>			1	
						 		-	
					 \	 		/	

LOG BOOK
Project: EPTC Ambient
Project #: C96-035

	Log Number	Sample ID	Date	Time	Comments	o = 0		st cloudy aken by	
	ρυ'	ARO · 3	10-15	12 <i>00</i> 1130	ARB COLEXICO	ζ		KEL	
	21	14-3	10-15	127C 1150	Heber Fire Department				
	<mark>አ</mark> ጉ	Mv-3	10-15	1240	megdans Union School				
	23	<u>E</u> L-3	10-15	1255 1030	El centio				
	24	「トーイエ	10-15	1310 1450	Imperial High				
	25	EL-YN	167-D	1600					
	26	EL-4B	10-17	1030 1000					
	27	IH-44	10-17	1020					
	78	IHYB	10-16	1050					
	<u>٦</u> ٩	MU 44	10-15	1045					
	3ნ	111-43	10-17	1115 1045	10-17 Paper Sample. Brice open				
	31	ARBUYA	10-16	1130 1100					
o ∕iâ	3)	ARBYIS	10-17	1130					
	33	144A	10-16	1150 1130.	·				
	34	H-413	10-17	1150 1132					
	35	EL-S&	10-18	1000 720					
	36	IH-5	10-14	1030 -735					
	37	MU-5	10-17	1045 0755					
	38	ARD-5	10-17	1100 0830					
	39	H-5	10-17	5310 5310	01130				
	40	6-5	10-14	©100	Blank	4	_	<u> </u>	
	L	<u></u>						•	

LOG BOOK
Project: EPTC Ambient
Project #: C96-035

Log Number	Sample ID	Date	Time	Comments	weather o = overce pc = parti k = clear	y cloudy
41	IH-6	10/22	11:1 3		K/K	exi
41	MU-6	10/12	11:49 11:06		K/K	
43	ARB-6	N/U N/U	12:L1 11:28		k/k	
44	н-6	10/22	12:39 11:49		K/K	
45	EL-6	10/21	B :13 10:7		KK	
46	EL-7	10/12 10/13	1017 927		K/K K/PC K/PC K/PC K/PC	
47	IH-7	10/23	1042 C944		KIPC	
48	MU-7	10/23	110h		K/PC	
49	ARB-7	10/22	1040		K/PC	
50	µ-7	10/22 10/13	11:49		14/	
51	EL-8A	10/13	C930	20t. # 2A	PC/PC	
52	EL-8B	10/13 10/14	0930	Dup. Rub # 28	PC/PC	
<i>5</i> 3	IH-8A	10/24	0944	Zut# 3A	PC/PC PC/PC	
54	IH-8B	10/13	0944	. Dup. Rut. # 3B	PC/PC	
55	MU-8A	10H3	1009	Rot. # 1A	PC/PC	
56	MU-8B	10/14	1009	Dup. Rot. # 113	MAC	
57	ARB-8A	10/23	1040	Rot. #4A	Pr	
58	ARG-8B	10/23	1040	Dup. Rot. #4B	PC	
<i>5</i> 9.	H-8A	10/123 10/124 10/123	1108		PC/PC PC/PC PC/K	
60	H-8B	10/24	110x 1135 0930	Dup.	PCI	
61	EL-9	10/15	0900		PCI	
62	IH-9	1915	0130		PSK	\

Project: EPTC Ambient Project #: C96-035

Log Number	Sample ID	Date	Time	Comments	weather o = overca pc = partly k = clear	cloudy
63	MU-9	10/14	1023 1035		PC/K	EXL
64	ARB-9	ार्गि १०१६५	1244 1139		PCK	
65	H-9	10/15	1135		PC/K	
66	B-9 Blank	10/24	\angle			
67	EL-10	10/25	900 - 730		K/20	
68	IH-P	10/25	0851		K/PC K/PC K/PC K/PC PC/PC PC/PC PC/PC PC/PC PC/PC PC/PC	
69	MU-10	10/25	1035 0915		K/AL	
70	ARB-10	10/26	1109 0933		K/PC	
71	<u>ij-10</u>	10/15	100%	Sampler fell down I high wind	R/PC	
72	EL-11	1-127	0830 0813		PC	
73	IH-11	10/20	1560		PC/PC	
74	MU-11	10.27	की5 ०४%		Pck	
15	ARB-11	10/26	9733 2914		PC	
76	H-11	10/16	100X 1938		PC/PC	
77	EL-12A	10/27	7813 7647	Rot. #ZA	Pc/pt	
78	EL-11B		07/3 7/f2	Rot. # ZB Dup.	PC/FE	
79	IH-12A	10/27	0831	Rot. # 3.4	PL	
80	IH-123	10/27	0831	Rot. # 3B Dup.	Pro	
81	MU-12A	10/27	0855		PC/PC PC/PC PC/PC PC/PC	
28	MU-12B		0731		PC	
83	ARB-NA		२१।५ ८४४ ०१।५	, · · · · · · · · · · · · · · · · · · ·	PC	
34	ARB-RB	10/27	0000		' IPC	*

LOG BOOK
Project: EPTC Ambient
Project #: C96-035

Log Number	Sample ID	Date	Time	Comments	weather o = overce pc = parti k = clear	y cloudy
85	H-12A	10/27 10/27	0938		PC/PC	EXL
86	H-12B	19121	0730		Pe/pc	
87	EL-13	12/27	2741		p'c/pc	
88	IH-13	10/18	-711 1806		PC/PC	
79	MU-13	10128	2731 0831		PS/PC	
90	ARB-13	13/28	0752		PYPC	
91	H-13	10/73	0750		Pelpe Pelpe Pelpe Pelpe Pelk Pelk Pelk Pelk	
92	EL-14	12/12	0741 -655		PC/K	
93	TH-14	10/19	0706 07/2		PC/K	
94	MU-H	1429	0831		PC/K	
95	ARB-14	10130	0952		PUK	
96	H-14	19,29	0837		PC/K	+
17	B-14 Blank	Heap	4940			
98	EL-15	11-4	1346			
99	H-15	11-4	1415 1200			
,DO	iARB-15	11-4	1225			
101	M4-15	11-4	1505 1300 1525			
102	I#-15	11-5	1330			
103	EL-16	11-5	//30 // <i>0</i> 0			
104	SPICFI	11-5	1130 1100 1200	Spike		
105	H-16	11-5	1130			
106	EPTC-FZ	11-5	1200	Spike		

LOG BOOK
Project: EPTC Ambient
Project #: C96-035

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear taken by
107	ARB-16	11.5	1225		
108	EPTC F3	11-62	1225	Spike	
109	MUIL	11-5	1150 1300 1215		
110	EPTC FY	11-5	1300	Spike Spike	
		11-6	125	Spi Re	
	IH-16	11-6	1300	Spike	
12	EPTC F3	11-6	/3∞ 1 100	Spike	
113	EL-17	11-7	0930		
119	EL-17	11-4 11-7	11 6 0 (530	Dup	
115	H-17	11-6	1000	•	
liv	B H-17	11-6	1000	Dup	
117	ARB-17	11-6	1150		
118	6	11-6	1150		
	ARB-17	11-6	1020	Dup	
119	Mu-17	11-7	1050		
120	Mu-17	11-7	105	Dup	
121	IH-17	11-9	1300		
122	IH-17	11-6	1300	Dup	
123	EC-18	11-7	0930		
124	H-18	11-7	1000 0700		
125	ARB-18	11-7	1020		
		11-8	0925 1050		
126	mu-18	11-8	1000		
127	IH-18	11-8	1030		
158	BLAUK	16	1001		

Project: EPTC Ambient Project #: C96-035

	Log Number	Sample ID	Date	Time	Comments		verca partly	st cloud taken	
129	H-19		11/12/96	1425		K	,	XE	M
'			11/12/96	1320	1500	1	`		
136	ARB-M		11/13	1400)	
171	50 C		11/12/16	1400	·	1 1		- 1	İ
131	Mu-19		1///	1340		├			
132	IH-19		11112196	1340				ļ	1
	ا ا - الض		11/12/46	1320		1			-
133	EL-19		11/13/96	1300		1 1		1	
1			11/12	1320					
134	F5/A19		11/13	1300					
125	•		11/12	1320		1 1			
135	ZA 19		11/13	1360		1			
136	3A 19		ii/n	1326			/		
150	ון אנ		11/13	1300		- · · ·			\vdash
137	EL 20		1/19	1130		人	/		
1			11/13	1306		1			\Box
138	El 200		11/14	1130		<u> </u>			
		- il -	11/13	1320		1 /			
	139	IH 20	11/14	1200		1			$\vdash \vdash$
	140	+110-1	11/13	1320		1 1			1 1
		IH200	11/14	1340	<u></u>	 			
	141	MU20	11/14	1230					i
		FIUZU	11/13	1340		1			\Box
	142	MUZON	11/14	1230	<u>.</u>				
			11/13	1400					
	143	ARB20	11- 14	1315		ļ			
			11/13	1400					l
	144	AKK200	1 - 1 - 1	13/5		ļ			
	1111	HZO	1/13	1430					
	145	1120	1//13	1245		 			\dashv
	146	HZOD	11/14	1245					
			11/19	1130		1			
ļ	147	EL21	11/15	0200	<u> </u>				
	1. 0		11/14	11/30		/		1	
	148	FS 214A		<i>5</i> 9 - 6 0 • 0		 			
1	149	667121	11/14	1130		1 1			
İ	177	F5715A	11/14	1200		+	, 		$\overline{}$
	150	IH21	1//15	5130		1	/	V	_

151 MUZI 11/19 12:33 152 1721 11/19 12:45 153 NR21 11/19 12:15 Project -C96-045-035

Telone, Kern-Co. ambient

			<u> </u>	<u> </u>	
log number	sample ID	date	time	comments	taken by
164	EL-22	11/18	10:52		KIPC EXL
155	EL-22	11/18	10:38	Spike #1 5 pl	K/PC
156	EL-22	11/18	10:52	Spike #3 Sul	K/PC
157	EL-22	11/18 기기 기기	시년 기년 11:28	Trip Spike #4 5 jul	K/PC
158	1H-22	11/18	11:03		KI PC
159	MU-22	11/18	11:25		K/PC
160	ARB-22	11/19	12:05		K/PC
<u>i6!</u>	H-22	1/19	11:44		KIPC
167	EL-23	11/19	7:49		Prik Colon
163	EL-23	11/19	7.49 12.38	Spike #5 10 pul Spike #6 10 pul	PC/K
164	EL-23	41149	7:49 N/A	Trip Spike #7 10jul	
166	FL-23 IH-23	N/A 11/19 11/20	N/A //:03 8:39	THE SPACE WILLIAM	PC/K
167	MU-23	11/19	11:25		PC/IL
11.8	H-23	11/19	11:74		PC/k
169	ABB-23	11/19	12:05		PC/K
170	EL-24	11/20	7:49		KIRC
17/	EL-24	11/10	7.49	Duplicate	K/PC
m	1H-24	11/43	8:39		K/H
173	1H-24	11/20	6.57	Duplicate	K/PC
174	MU-24	11/20	7:16		K/Tt
115	MU-24	11/11	7:16	Ouplicate	KIPC

LOG BOOK
Project C96-045-035
Telono, Kern Go. ambient

EPTC										
log number	sample ID	date	time	comments		taken by				
171.	ARB-24	11/20	9.20	·	K/Pc					
177	ARB-24	11/21	7:35	Duplicate	KIPC					
178	H-24	11/12 11/12 11/12	9.45 7.16 9.45		K/R					
173	H-24	/1/21 NIA	876 NIA	Duplicate	KIPC					
130	Blank	NIA NIA	NIA	Duplicate Sample Blank Blank (Trip) for Spikes						
181	Blank	~/A	MA	19lank (Trip) for Spikes		<u>. </u>				
					-					
	·									
					-	*				
					1					

B24A B24B

APPENDIX VII APPLICATION METEOROLOGICAL DATA

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

			Wind		Barometric	III. avela	Wind
	Julian		Speed	Temp.	Pressure	Relative	Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	(degrees)
1997	146	1227	8.53	78.6	1013	30.45	318
1997	146	1243	10.98	80.2	1013	26.85	322
1997	146	1258	7.56	81.3	1013	28.18	307
1997	146	1313	5.96	81.6	1013	27.83	330
1997	146	1328	7.17	82.5	1013	26.24	341
1997	146	1343	5.00	83.4	1013	25.15	342
1997	146	1358	8.36	83.8	1013	19.78	304
1997	146	1413	8.75	84.1	1013	19.06	345
1997	146	1428	12.10	84.2	1013	19.18	317
1997	146	1443	9.93	84.3	1013	20.40	341
1997	146	1458	10.47	84.1	1013	24.27	337
1997	146	1513	5.62	84.3	1013	27.82	340
1997	146	1528	4.74	83.9	1013	27.86	329
1997	146	1543	3.70	84.3	1013	29.89	333
1997	146	1558	3.50	83.3	1013	32.75	328
1997	146	1613	5.24	83.9	1013	37.95	331
1997	146	1628	10.45	82.2	1013	42.70	326
1997	146	1643	12.31	81.5	1013	46.47	316
1997	146	1658	9.06	81.3	1013	47.62	313
1997	146	1713	14.39	80.6	1013	48.05	323
1997	146	1728	12.11	80.4	1013	49.57	319
1997	146	1743	13.02	80.4	1013	51.00	324
1997	146	1758	15.15	79.4	1013	49.76	328
1997	146	1813	12.27	78.6	1013	52.56	331
1997	146	1828	11.96	78.4	1013	51.23	331
1997	146	1843	12.72	77.9	1013	51.62	332
1997	146	1858	13.75	78.0	1013	49.64	322
1997	146	1913	11.33	77.3	1013	54.30	329
1997	146	1928	9.25	75.2	1013	61.89	330
1997	146	1943	11.07	74.1	1013	64.54	336
1997	146	1958	5.54	73.4	1013	62.99	334
1997 1997	146 146	2013	1.31	72.3	1013	65.70	332
1997		2028 2043	1.02	71.4	1013	68.64	330
1997	146 146	2043	0.15	70.7 69.6	1014 1014	69.56	335
1997	146	2113	0.00			72.83	331
1997	146	2113	0.00	69.8 69.6	1014 1014	73.24 73.43	330 333
1997	146	2143	0.00	69.4	1014	73.43	333
1997	146	2143	0.00	69.3	1014	74.77	327
1997	146	2138	0.13	69.0	1014	75.69	323
1997	146	2228	2.11	68.2	1014	77.61	319
1997	146	2243	2.62	67.9	1014	79.16	320
1997	146	2258	6.21	68.3	1014	75.64	327
1997	146	2313	7.47	68.9	1014	74.78	331

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

			Wind		Barometric		Wind
	Julian		Speed	Temp.	Pressure	Relative	Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	(degrees)
1997	146	2328	7.15	69.4	1014	75.57	326
1997	146	2343	6.85	69.6	1015	74.41	329
1997	146	2358	6.34	69.4	1015	76.13	330
1997	147	13	7.59	69.1	1015	79.59	327
1997	147	28	9.26	68.9	1014	84.37	325
1997	147	43	8.57	68.8	1014	87.21	332
1997	147	58	6.75	68.1	1014	85.57	338
1997	147	113	6.06	67.7	1014	86.39	239
1997	147	128	7.16	67.3	1014	82.50	123
1997	147	143	7.07	67.1	1015	85.73	011
1997	147	158	8.33	66.9	1014	86.02	147
1997	147	213	8.73	66.7	1014	86.20	261
1997	147	228	8.84	66.5	1014	87.33	260
1997	147	243	8.13	66.3	1014	89.11	172
1997	147	258	7.79	65.8	1015	89.51	123
1997	147	313	6.16	65.1	1015	90.13	191
1997	147	328	3.78	64.3	1015	91.11	286
1997	147	343	2.08	63.8	1015	92.56	193
1997	147	358	5.69	64.0	1015	92.61	218
1997	147	413	2.35	63.6	1015	93.09	304
1997	147	428	0.12	62.7	1015	94.75	327
1997	147	443	0.83	63.1	1015	93.53	283
1997	147	458	0.67	62.7	1015	94.89	347
1997	147	513	0.44	62.3	1015	95.59	341
1997	147	528	0.03	62.2	1015	95.31	298
1997	147	543	0.00	61.5	1015	96.83	282
1997	147	558	0.00	61.4	1015	97.03	290
1997	147	613	0.10	61.1	1015	98.24	312
1997	147	628	0.00	61.3	1015	98.49	334
1997	147		0.34	62.0			
1997	147	658	0.75	62.6	1015	97.39	305
1997	147	713	0.38	63.5	1015	96.26	319
1997	147	728	2.29	64.7	1015	93.83	309
1997	147	743	5.71	65.7	1015	90.79	312
1997	147	758	6.67	67.1	1015	87.23	310
1997	147	813	8.62	68.5	1015	83.77	318
1997	147	828	10.25	69.9	1016	80.31	322
1997	147	843	11.56	70.7	1016	77.19	326
1997	147	858	11.48	71.5	1016	75.25	332
1997	147	913	11.43	72.1	1015	73.82	
1997	147	928	11.43	72.5	1015	72.49	
1997	147	943	10.44	73.3		71.52	337
1997	147	958	11.05	74.1	1015	70.06	
1997	147	1013	11.11	74.9	1015	68.21	330

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

	Julian	İ	Wind Speed	Tomp	Barometric Pressure	Relative	Wind Direction
Year	Date	Time	(mph)	Temp. (F)	(hPa)	Humidity	(degrees)
1997	147	1028	12.78	75.3	1015	66.59	328
1997	147	1043	12.45	75.5	1015	65.29	326
1997	147	1058	12.38	76.6	1014	63.65	328
1997	147	1113	13.19	76.6	1014	63.10	324
1997	147	1128	12.45	77.3	1014	62.38	330
1997	147	1143	12.73	78.0	1014	61.60	324
1997	147	1158	12.08	78.9	1014	59.50	336
1997	147	1213	12.45	79.4	1014	59.15	310
1997	147	1228	13.00	79.7	1014	58.57	334
1997	147	1243	12.45	80.2	1014	58.55	311
1997	147	1258	13.88	80.8	1014	56.81	320
1997	147	1313	12.66	81.1	1014	56.49	331
1997	147	1328	13.95	82.1	1013	54.07	341
1997	147	1343	13.10	82.6	1013	51.46	334
1997	147	1358	13.70	82.5	1013	49.99	337
1997	147	1413	14.23	83.2	1013	50.21	335
1997	147	1428	14.50	83.5	1013	48.58	335
1997	147	1443	13.85	84.2	1013	46.61	346
1997	147	1458	14.16	84.7	1013	47.57	317
1997	147	1513	13.69	85.0	1013	45.69	343
1997	147	1528	10.04	85.5		45.22	325
1997	147	1543	6.75	85.5	1012	45.94	348
1997	147	1558	1.34	86.2	1012	45.94	324
1997	147	1613	0.85	85.9	1012	46.01	343
1997	147	1628	0.49	86.4	1012	46.00	343
1997	147	1643	0.33	86.1	1012		320
1997	147	1658	0.00	86.9		44.99	
1997	147	1713	0.20	86.2		46.24	350
1997	147	1728	0.02	86.0		47.09	
1997	147		0.01	85.9		48.08	
1997	147	1758	0.06	85.8		48.52	
1997	147	1813	0.02	85.8	<u> </u>	48.27	322
1997	147	1828	0.66	85.4		48.82	
1997	147	1843	0.52	85.2		47.95 49.28	
1997	147	1858	0.74 0.00	84.8 84.0		52.15	
1997	147 147	1913 1928	0.00	81.0	1		
1997 1997	147	1928	0.00	79.4		59.81	332
1997	147	1943	0.00	77.8		62.51	320
1997	147	2013	0.00	77.0 75.9		<u> </u>	
1997	147	2013	0.00	74.6			
1997	147		0.00	73.5			
1997	147		0.00			<u> </u>	
1997	147			<u> </u>		1	

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

			Wind		Barometric		Wind
!	Julian		Speed	Temp.	Pressure	Relative	Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	
1997	147	2128	0.00	71.3	1011	73.37	327
1997	147	2143	0.00	70.6	1011	73.14	293
1997	147	2158	0.00	71.0	1012	70.57	328
1997	147	2213	0.00	70.5	1011	70.95	328
1997	147	2228	0.00	70.5	1011	70.75	
1997	147	2243	0.00	70.2	1011	71.66	
1997	147	2258	0.00	70.2	1011	71.40	
1997	147	2313	0.00	68.3	1011	76.56	1
1997	147	2328	0.00	67.6	1011	77.99	
1997	147	2343	0.00	66.5	1011	81.22	
1997	147	2358	0.00	66.3	1011	80.82	<u> </u>
1997	148	13	0.00	65.9	1011	80.79	
1997	148	28	0.00	66.2	1011	79.67	<u> </u>
1997	148	43	0.00	66.4	1011	79.13	
1997	148	58	0.00	67.3	1011	77.17	1
1997	148	113	0.00	68.2	1011	74.83	
1997	148	128	0.00	67.4	1011	77.36	<u> </u>
1997	148	143	0.00	69.0	1011	73.95	
1997	148	158	0.07	66.6	1011	79.61	
1997	148	213	1.04	65.4	1011	82.75	
1997	148	228	1.77	66.3	1011	81.51	
1997	148	243	0.00	65.3	1011	85.91	
1997	148	258	0.00	64.5	1011	87.03	
1997	148	313	0.00	64.8	1011	86.63	
1997	148	328	0.00	65.5	1011	85.09	
1997 1997	148 148	343 358	0.00	64.5	1011	86.93 87.59	<u> </u>
1997	148	413	0.00	64.1 63.9	1011	89.15	
1997	148	413		62.4	1011	92.11	
1997	148		0.00				236 258
1997	148	443	0.00	61.4 59.2	1011	93.45 96.99	
1997	148	513	0.00	59.2	1011	98.77	
1997	148	528	0.00	59.5		97.47	
1997	148	543	0.00	59.5	1011	96.66	
1997	148	558	0.00	59.7	1011	96.24	
1997	148	613	0.00	60.6	1011	95.63	L
1997	148	628	0.00	61.6	1011	94.77	
1997	148	643	0.00	61.9	1011	96.15	
1997	148	658	0.00	63.9	1011	94.22	
1997	148	713	0.00	66.0	1011	92.07	<u> </u>
1997	148	728	0.00	67.7	1011	88.64	
1997	148	743	0.00	68.4	1012	86.07	
1997	148	758	0.00	70.4	1012	82.53	
1997	148	813	0.04	72.3	1012	78.92	298

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

	Julian		Wind Speed	Temp.	Barometric Pressure	Relative	Wind Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	(degrees)
1997	148	828	0.34	73.0	1012	75.11	194
1997	148	843	0.00	73.9	1012	73.17	086
1997	148	858	0.00	75.8	1012	71.32	180
1997	148	913	0.00	76.5	1012	69.95	217
1997	148	928	0.00	79.0	1012	67.86	211
1997	148	943	0.09	78.8	1012	65.33	224
1997	148	958	0.00	78.4	1012	65.33	320
1997	148	1013	0.00	80.1	1011	63.96	286
1997	148	1028	0.11	81.3	1011	61.43	320
1997	148	1043	0.00	82.1	1011	59.46	324
1997	148	1058	0.46	82.3	1011	57.88	321
1997	148	1113	0.31	82.9	1011	57.71	330
1997	148	1128	0.28	84.0	1011	57.47	333
1997	148	1143	0.60	84.9	1011	55.89	333
1997	148	1158	0.98	85.5	1011	53.67	332
1997	148	1213	0.87	86.1	1011	52.59	312
1997	148	1228	0.23	86.3	1011	52.84	311
1997	148	1243	0.01	86.9	1011	51.02	334
1997	148	1258	0.00	88.0	1011	49.30	331
1997	148	1313	0.01	88.3	1010	48.74	316
1997	148	1328	0.00	88.5	1010	47.27	337
1997	148	1343	0.16	88.9		45.66	
1997	148	1358	0.00	88.7	1010	45.83	
1997	148		0.00	89.6			
1997	148	1428	0.00	90.3			
1997	148		0.00	90.5		41.96	
1997	148	1458	0.01	90.5		41.90	
1997	148	 	0.00	90.7		42.53	<u> </u>
1997	148		0.00	89.5	<u> </u>	45.05	<u> </u>
1997							
1997	148		0.01	88.0	<u> </u>		
1997	148		0.18				
1997	148		1.51	89.1			
1997	148		0.60	87.6			<u> </u>
1997	148		0.68	85.9			4
1997			0.42				
1997			3.90	84.6			
1997 1997	1	<u> </u>	5.56 8.90				<u> </u>
1997			7.48			1	
1997			2.96				
1997			6.64				<u></u>
1997	} 				 		
1997	<u> </u>					<u> </u>	
1997	148	1913	3.25	02.3	1008	35.76	34,

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humiditý	Wind Direction (degrees)
1997	148	1928	2.51	81.0	1008	57.37	
1997	148	1943	0.35	79.3	1008		344
1997	148	1958	0.00	78.8	1008		L
1997	148	2013	0.00	78.0	1008		
1997	148	2013	0.02	77.0	1008		
1997	148	2028	0.00	75.7	1008		
1997	148	2043	0.00	74.9	1008	67.42	
1997	148	2113	0.00	73.5	1008		
1997	148	2128	0.00	72.5			<u> </u>
1997	148	2143	0.00	71.7	1008	73.93	
1997	148	2158	0.00	70.0	1009	77.52	
1997	148	2213	0.00	68.2	1009	81.39	<u> </u>
1997	148	2228	0.00	68.1	1009		
1997	148	2243	0.00	69.6	1009		268
1997	148	2258	0.00	69.6	1009	75.45	
1997	148	2313	0.00	69.0	1009		
1997	148	2328	0.00	67.1	1009		
1997	148	2343	0.00	66.6	1009	79.63	
1997	148	2358	0.01	66.4	1009	79.59	
1997	149	13	0.00	65.8	1009		
1997	149	28	0.00	66.2	1009	79.72	021
1997	149	43	0.00	65.8	1009	80.47	189
1997	149	58	0.00	65.2	1009		
1997	149	113	0.00	65.1	1009	82.37	322
1997	149	128	0.00	64.5	1009		
1997	149	143	0.00	65.3	1009	82.51	068
1997	149	158	0.00	64.6	1009		
1997	149	213	0.00	63.7	1009		
1997	149	228	0.00	63.8	1009	88.54	
1997	149		0.00	64.3			
1997	149	258	0.00	64.4	1009	86.17	234
1997	149	313	0.00	64.6	1009	86.10	264
1997	149	328	0.00	64.6	1009	87.09	
1997	149	343	0.00	63.4	1008		
1997	149	358	0.00	62.0	1008		
1997	149	413	0.00	62.2	1008	91.01	187
1997	149	428	0.00	62.8	1008	90.29	215
1997	149	443	0.00	62.2	1008	91.81	222
1997	149	458	0.00	62.3	1008	92.51	236
1997	149	513	0.00	62.0	1008	92,19	244
1997	149		0.00	61.6	1008	91.83	
1997	149		0.00	62.0	1008	91.84	
1997	149		0.00	62.3	1008		
1997	149	613	0.00	61.4	1009	92.62	232

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

	Wind Barometric						Wind
1	Julian		Speed	Temp.	Pressure	Relative	Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	(degrees)
1997	149	628	0.00	61.1	1009	95.23	240
1997	149	643	0.00	63.5	1009	93.32	250
1997	149	658	0.00	65.7	1009	91.95	150
1997	149	713	0.00	66.7	1009	89.79	146
1997	149	728	0.00	68.4	1009	86.52	251
1997	149	743	0.00	67.3	1009	87.45	278
1997	149	758	0.00	68.8	1009	83.47	285
1997	149	813	0.00	71.0	1009	80.31	275
1997	149	828	0.00	73.3	1009	77.27	303
1997	149	843	0.00	74.6	1009	73.95	297
1997	149	858	0.01	75.9	1009	69.69	291
1997	149	913	0.06	77.6	1009	65.71	309
1997	149	928	0.68	78.9	1009	62.42	327
1997	149	943	0.38	80.0	1009	61.86	304
1997	149	958	2.98	82.0	1009	58.27	326
1997	149	1013	1.15	83.3	1009	56.46	332
1997	149	1028	2.99	84.5	1009	53.35	326
1997	149	1043	4.15	84.9	1009	50.37	325
1997	149	1058	4.36	85.5	1009	48.95	322
1997	149	1113	3.04	86.0	1009	48.61	328
1997	149	1128	2.90	86.7	1009	48.58	
1997	149	1143	1.95	87.5	1008		
1997	149	1158	0.64	87.7	1008		
1997	149	1213	0.05	88.4	1008		
1997	149	1228	0.00	89.7	1008		
1997	149	1243	0.01	89.7	1008		
1997	149	1258	0.00	90.6			
1997	149	1313	0.00	90.8			
1997	149	1328	0.00	91.4	<u> </u>		1
1997	149						
1997	149	1358	0.00	91.8		I	
1997	149	1413	0.00	92.3			
1997	149	1428	0.00	92.8			
1997	149	1443	0.00	93.2			
1997	149	1458	0.00	93.5			
1997 1997	149 149	1513 1528	0.00	93.2 92.4			
1997	149	1543	0.00	91.2			<u> </u>
1997	149	1558	0.00	90.8	<u> </u>		
1997	149		0.00	91.9	1	<u></u>	1
1997	149		0.00	91.2		1	
1997	149		0.00	90.5			<u> </u>
1997			0.00				
1997			0.00				

EPTC APPLICATION METEOROLOGICAL DATA (15 min. averages)

			Wind		Barometric		Wind
	Julian		Speed	Temp.	Pressure	Relative	Direction
Year	Date	Time	(mph)	(F)	(hPa)	Humidity	(degrees)
1997	149	1728	0.00	92.8	1006	37.15	336
1997	149	1743	0.00	92.7	1006	37.34	342
1997	149	1758	0.00	92.4	1006	38.56	
1997	149	1813	0.00	91.9	1006	39.24	335
1997	149	1828	0.00	91.0	1006	39.69	
1997	149	1843	0.00	90.2	1006	40.86	333
1997	149	1858	0.00	89.3	1006	41.85	334
1997	149	1913	0.00	88.3	1006	40.98	343
1997	149	1928	0.00	86.7	1006	43.86	339
1997	149	1943	0.00	84.6	1006	49.30	331
1997	149	1958	0.00	82.4	1007	50.62	331
1997	149	2013	0.00	80.2	1007	52.76	334
1997	149	2028	0.00	78.5	1007	54.54	337
1997	149	2043	0.00	77.1	1007	55.90	342
1997	149	2058	0.00	76.3	1007	55.87	343
1997	149	2113	0.00	75.7	1007	56.12	339
1997	149 149	2128 2143	0.00	74.4	1007	58.57	332
1997 1997	149	2143	0.00	73.0	1007	60.87	328
1997	149	2213	0.00	72.1 70.7	1007	62.12	319
1997	149	2213	0.00	70.7	1007 1007	65.45 65.25	321 334
1997	149	2243	0.00	69.9	1007	66.00	322
1997	149	2258	0.00	68.2	1007	69.60	312
1997	149	2313	0.00	68.5	1007	66.65	304
1997	149	2328	0.00	68.6	1007	67.43	340
1997	149	2343	0.00	67.3	1007	70.96	295
1997	149	2358	0.00	68.8	1007	65.04	309
1997	150	13	0.00	68.3	1007	66.50	325
1997	150	28	0.00	68.4	1007	65.63	066
1997	150		0.00		1007		
1997	150	58	0.00	68.0	1007	69.15	
1997	150	113	0.00	68.0	1007	70.62	
1997	150	128	0.00	67.7	1007	71.09	
1997	150	143	0.00	67.1	1007	72.57	065
1997	150	158	0.00	66.8	1007	73.94	213
1997	150	621	0.00	65.8	1007	80.24	289
1997	150	636	0.00	65.5	1008	90.13	186
1997	150	651	0.00	68.7	1008	85.21	260
1997	150	706	0.00	69.3	1008	80.97	323
1997	150	721	0.00	69.3	1008	79.45	326
1997	150	736	0.00	68.6	1008	76.74	228
1997	150	751	0.00	69.9	1008	73.00	249
1997	150	806	0.00	71.7	1009	68.40	127

APPENDIX VIII STANDARD OPERATING PROCEDURE

State of California Air Resources Board Monitoring and Laboratory Division/ELB

Standard Operating Procedure for the Sampling and Analysis of EPTC in Ambient Air

1. SCOPE

This is a sorbent tube, solvent extraction, gas chromatography/mass selective detector method for the determination of EPTC from ambient air samples.

2. SUMMARY OF METHOD

The exposed XAD-2 resin tubes (SKC #226-30-06) are stored in an ice chest on dry ice or freezer until desorbed with 3 ml of ethyl acetate. The splitless injection volume is 2 ul. A gas chromatograph with a DB-35 capillary column and a mass selective detector are used for analysis.

3. INTERFERENCES/LIMITATIONS

Method interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. A method blank must be done with each batch of samples to detect any possible method interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett Packard 5890 chromatograph Hewlett Packard 5971A mass selective detector Hewlett Packard Varian 8200 Autosampler

Detector: 250°C Injector: 250°C

Column: J&W Scientific DB-35, 30 meter, 0.25 mm i.d., 0.1 um film thickness.

GC Temp. Program: Initial 60°C, hold 2 min, to 150°C @ 15°C/min., to 170 °C @ 5°C/min., to 250 °C @ 70 °C/min., hold 5 min.

Gas Flows: column: He, 1.0 mL/min (8.0 psi @ 60 °C), electronic pressure control

B. AUXILIARY APPARATUS:

- 1. Glass amber vials, 8 mL capacity.
- 2. Vial Shaker, SKC, or equiv.
- 3. Autosampler vials with septum caps.

C. REAGENTS

- 1. Ethyl Acetate, Pesticide Grade, or better
- 2. EPTC, 98% pure or better (e.g., from Chem Service).
- 3. Biphenyl D-10, 98% pure or better (e.g., from Cambridge Isotope Laboratories)

5. ANALYSIS OF SAMPLES

- 1. It is necessary to analyze a solvent blank with each batch of samples. The blank must be free of interferences. A solvent blank must be analyzed after any sample which results in possible carry-over contamination.
- 2. If a standard curve is not generated each day of analysis, at least one calibration sample must be analyzed for each batch of ten samples. The response of the standard must be within 10% of previous calibration analyses.
- 3. Carefully score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug from the primary end of the XAD-2 tube with forceps and place it into an 8 mL amber colored sample vial. Pour the XAD-2 into the vial and add 3 mL ethyl acetate. Retain the secondary section of the XAD-2 tube for later analysis if needed to check the possibility of breakthrough.
- 4. Place the sample vial on a desorption shaker (or ultra sonic water-bath) for 30 minutes. Remove the EPTC extract and store in a second vial at -20°C until analysis.
- 5. After calibration of the GC system, inject 2.0 ul of the extract. If the resultant peaks for EPTC have a measured area greater than that of the highest standard injected, dilute the sample and re-inject.
- 6. Calculate the concentration in ng/mL based on the data system calibration response factors. If the sample has been diluted, multiply the calculated concentration by the dilution factor.
- 7. The atmospheric concentration is calculated according to:

Conc., ng/m³ = (Extract Conc., ng/mL X 3 mL) / Air Volume Sampled, m³

6. QUALITY ASSURANCE

A. INSTRUMENT REPRODUCIBILITY

Triplicate injections of 2 uL each were made of EPTC standards at five concentrations in order to establish the reproducibility of this instrument. This data (Testing Section lab, 9/30/96) is shown in Table 1.

TABLE 1 Instrument Reproducibility

I.S. Amt. (ng/ml)	I.S. Response (area counts)	EPTC Amt. (ng/ml)	EPTC Response (area counts)	Amt. Ratio	Resp. Ratio	Response Ratio RSD
500	53662	111	3605	0.222	0.0672	
500	52463	1,11	3445	0.222	0.0657	
500	52421	111	3457	0.222	0.0659	1.2%
500	50775	223	6751	0.446	0.133	
500	49663	223	6679	0.446	0.135	
500	50170	223	6666	0.446	0.133	0.9%
500	50018	446	12644	0.892	0.253	
500	49995	446	12702	0.892	0.254	
500	49751	446	12707	0.892	0.255	0.4%
500	49961	891	27368	1.78	0.548	
500	51557	891	27965	1.78	0.542	
500	51237	819	27757	1.78	0.542	0.6%
500	50653	1782	55400	3.56	1.09]
500	52334	1782	56653	3.56	1.08	
500	51666	1782	55698	3.56	1.08	0.5%

B. LINEARITY

A five point calibration curve was made (Testing Section lab, 9/30/97) ranging from 111 ng/mL to 1782 ng/mL EPTC (from TABLE 1). The corresponding linear regression equation and correlation coefficient are:

Response Ratio = (0.306)(Amount Ratio) - 0.00628 Corr. Coef. = .997

where:

Response Ratio = (EPTC response)/(Biphenyl D-10 response)

Amount Ratio = (EPTC concentration)/(Biphenyl D-10 concentration)

C. MINIMUM DETECTION LIMIT

Using the equations above, the limit of detection (LOD) and the limit of quantitation (LOQ) for EPTC were calculated by:

$$LOD = |A| + 3(S)$$

where:

|A| = the absolute value of the x-intercept of the standard curve (from above).

(S) = the relative standard deviation of the responses of the lowest concentration used for the standard curve times A (RSD x A).

$$LOD = |0.0205| + 3(0.0205)(0.0122) = 0.0213 =$$
(response ratio) = 10.6 ng/ml EPTC

$$LOQ = 3.3(LOD) = 35.1 \text{ ng/ml}$$

Based on the 3 mL extraction volume and assuming a sample volume of 2.69 m³ (1.87 lpm for 24 hours):

 $(35.1 \text{ ng/mL})(3 \text{ mL}) / (2.69 \text{ m}^3) = 39.1 \text{ ng/m}^3 \text{ per } 24\text{-hour sample}$

D. COLLECTION AND EXTRACTION EFFICIENCY (RECOVERY)

Fifteen microliters of a 356.4 ng/ml EPTC standard were spiked on the primary section of each of four XAD-2 sampling tubes. Another set of 4 tubes were spiked with 7.5 microliters of the same standard solution. The spiked tubes were then subjected to an air flow of 2 lpm for 24 hours. The samplers were set-up in a garage/shop at an ambient temperature of approximately 90°F (maximum). The primary and back-up sections were then separately desorbed with ethyl acetate and

analyzed. One of the tubes spiked with 15 uL was broken during sample recovery. Percent recoveries from the primary sections of the three tubes spiked with 5346 ng (15 uL) EPTC were 84.5%, 96.8% and 89.6% with an average of 90.3%. Percent recoveries from the primary sections of the four tubes spiked with 2673 ng (7.5 uL) EPTC were 96.0%, 103%, 102% and 102% with an average of 101%.

E. STORAGE STABILITY

Storage stability studies were conducted over a 29 day period. The primary sections of eight tubes were spiked with 2673 ng of EPTC. The spiked tubes were stored in the freezer at -20 C and extracted/analyzed on storage days 0, 2, 6 and 29. Two tubes each were analyzed on each day. The storage recoveries (average results) were 101%, 110%, 110% and 109% for days 0, 2, 6 and 29 respectively.

F. BREAKTHROUGH

The primary sections of three tubes were spiked with 5346 ng EPTC/tube then run for 24 hours at 2 lpm (see Section D above). No EPTC was detected in the back-up resin bed of any of the three tubes.